ASSESMENT OF SPONTANEOUS HEATING OF SOME INDIAN COKING AND NON-COKING COALS

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY
IN
MINING ENGINEERING

BY

RAJDEEP LAKRA

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DEPARTMENT OF MINING ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY
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Under the guidance of
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National Institute of Technology Rourkela

CERTIFICATE

This is to certify that the thesis entitled **"ASSESMENT OF SPONTANEOUS HEATING OF SOME INDIAN COKING AND NON-COKING COAL"** submitted by **Sri Rajdeep Lakra** in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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ABSTRACT

The auto oxidation of coal ultimately leads to spontaneous combustion which is the major root cause for the disastrous of coal mine. It is a slow process and the heat evolved is carried away by air. This process of self heating of coal or other carbonaceous material resulting eventually in its ignition is termed as “spontaneous heating” or “auto oxidation”. The auto-oxidation of coal is a complex physico-chemical process which is accompanied by the absorption of oxygen, formation of coal oxygen complexes and their decomposition leading to the liberation of heat. Therefore the assessment for this combustion is very much necessary. It depends upon different characteristics and properties of coal. Coal mine fire is a major problem worldwide and has been a great concern both for the industry and researchers in this field. Majority of fires existing today in different coalfields are mainly due to spontaneous combustion of coal. . It has been a major problem in the leading producing coal countries like Australia, India and China. The spontaneous heating susceptibility of different coals varies over a wide range and it is important to assess their degree of proneness for taking preventive measures against the occurrence of fires to avoid loss of lives and property, sterilization of coal reserves and environmental pollution and raise concerns about safety and economic aspects of mining etc. Although much research has been done on the subject, a proper assessment of the spontaneous heating susceptibility of coal needs to be done so that mine operators are notified well in advance and plan the working properly. Therefore, the determination of susceptibility of coals to spontaneous heating and their classification is essential to plan the production activities and optimize coal mine production within the incubation period.

The project deals with the assessment of various parameters of coal with the spontaneous heating tendency of coal. Eight coal samples were collected from Mahanadi Coalfields Limited (MCL), Orissa of Coal India Limited and another eight samples were collected from the different mines such as northern coalfield limited (NCL), Madhya Pradesh ; Bharat coking coalfield limited (BCCL), Dhanbad ; Tisco ; IIsco, Dhanbad. The project deals with assessment of various parameters determined by experimental techniques. The intrinsic properties as well as susceptibility indices of the coal samples have historically been considered to be prediction of self heating liability. Many methods are based on the measurement of the oxidation rate and ignition temperature.

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CHAPTER – 1

INTRODUCTION

1.1 Background

Coal is one of the primary sources of energy, accounting for about 67% of the total energy consumption in the country. About 70% of coal production is used for power generation in India. Indian coal has high ash content and low calorific value. According to the 2010 Statistical Energy Survey, India had end 2009 coal reserves of 58600 million tonnes, 7.09% of the world total. India had 2009 coal production of 557.57 million tonnes, 6.2% of the world total.. India now ranks 3rd amongst the coal producing countries in the world. About 88% of the total coal production in the country is produced by various subsidiaries (a total of 390 mines) of Coal India Ltd. NCL Produced 69.62 million tons of coal in the year 2008-2009 and according to 2010-11 the production of MCL is 100.28 million tonnes (mt). In 2009-10, MCL's coal output stood at 104.08 million tonnes. The BCCL has achieved production of 17.962 mt as against 17.450 mt during the same period last year. The spontaneous heating of coal varies over a wide range and it is important to assess their degree of proneness for taking preventive measures against the occurrence of fires to avoid loss of lives and property, sterilization of coal reserves and environmental pollution and raise concerns about safety and economic prospects of mining (Tripathy, 2001). Different methods have been adopted by various researchers of the world to find out the susceptibility of coal to spontaneous heating. A number of experiments have been done for evaluating the spontaneous heating susceptibility of coal viz., Crossing point temperature method (Didari et.al., 2000), Wet oxidation potential method (Tarafadar et.al., 1989), Differential thermal analysis (Nimaje et.al., 2010), Flammability temperature (Nimaje et.al., 2010). This propensity to self heating of coal also determine the incubation period of coal seam, which decide the size of the panel to be formed, which is a most important safety measuring in mine planning. A number of approachment have been acquired over the years to assess the proneness of coal to spontaneous heating. It is therefore imperative that the planners of a mine determine in advance the spontaneous heating susceptibility of the seam to be mined so

that either the coal has been extracted before the incubation period. The methods used to assess the propensities of coals to spontaneous heating in the present study are Proximate analysis, Flammability temperature, Differential thermal analysis (DTA - TG), Wet oxidation potential, Calorific value.

1.2 **Objective of the project**

The objective of the project is to carry out comparision among all the coal samples of coking and non coking coal, the parameters of coal in order to find out the properties that influence spontaneous heating propensity of coal. The project was divided into the following parts – 2

- **Literature review –** Collection of all the past works done by various authors both national and international as well as earning information on the topic.

- **Sample collection and preparation –** Sixteen samples were collected from MCL, NCL, IISCO, BCCL, TISCO for the purpose of analysis and the samples were collected and developed as per the Indian Standards.
- **Experimentation –** The experimentation are divided into two stages:
    - Determination of intrinsic properties of coal – proximate analysis, calorific value
    - Determination of susceptibility indices of coal – wet oxidation potential, differential thermal analysis, flammability temperature.
- **Analysis –** compare the parameter of the different collected coal samples of various experimental techniques of various experiments were done, for finding out susceptibility of coal to spontaneous heating.

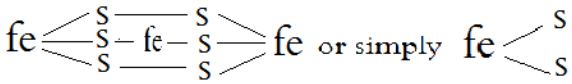
2.1 COAL MINE FIRES:

It is associated with mostly coal mines, through fires in pyrite mines and are common happenings in coal mines but are rare in metal mines. An analysis of the causes of coal mine fires bring out that they may start either from an open fire initiate due to the heavy nature of coal. Mine fires can be induced either by spontaneous heating, explosion of gases, electrical failures and blasting. In coal mines the major reason of mine fire is spontaneous heating of coal. The process in which the coal catches fire automatically on coming in impinging with oxygen in the atmosphere without any outside source of fire which leads to mine fires is acknowledged as spontaneous heating of coal. The property of coal releasing heat when in

contact with o_2 of air and the poor thermal conductivity privileging heat accumulation, may give rise to the latter type of heating. The former types of fire from external means are known as E*xogenous fires* and the latter, due to self-heating characteristics of coal, is called *Endogenous fires* or spontaneous combustion. It is chiefly thought that the main cause of spontaneous heating is the self-oxidation of coal. Although the actual mechanism of coal oxidation is yet unknown, there are many theories put forward for account of the coal oxidation and combustion.

2.1.1 ENDOGENOUS FIRES:

Pyrite fire:- The iron ore of pyrite, having chemical formula FeS_2 known as iron pyrite is polysulphide of iron believed to be of the following structure:



Pure pyrite contains 46.37% Fe and 53.33% S and is chiefly used in the manufacturer of H_2SO_4 and sulphur. As with coal pyrite also reacts with oxygen of air at room temperature emancipating heat which under favorable conditions of heat aggregation gives rise to spontaneous fires. In fact, spontaneous heating phenomenon of pyrite was known much earlier than that of coal. Susceptibility to spontaneous heating of pyrites is much less than that of coal but it increases if carbonaceous materials are present in pyrites. There are cases when pyrites with 5 - 6 % C and 10 – 12 % S have caught fire due to spontaneous heating.

Endogenous heating timber:- Usnder extremely favorable conditions decayed timber may give rise to spontaneous heating believed to be mainly from bacterial origin.

2.1.2 EXOGENOUS FIRE:

Electricity is one of the important induces of mine fires. It may originate from over heating of machines, short circuiting, candles, electric bulbs, flames from fires or explosion while blasting and ignition of inflammable materials like timber, oil or wastes. At times squeezing of sulphide ores or fires from surface may also be the origin. But fires of incitive origin are not of much practical importance. Fires from inflammable gases as CH_4, which may occur in certain seams.

2.2 HISTORY OF COAL MINE FIRES

Self heating of coal leading to spontaneous combustion is the most important cause of fires in coal mines across the world (Mahadevan and Ramlu 1985). Self heating of coal can occur in

underground mines, opencast mines, coal stockpiles, transportation and during the disposal of wastes from coal using industries in heap wastes (Bowes, 1984; Carras and Young 1994).

World scenario:

In china underground coal fires are far-flung within a region elongating 5000 Km east – west and 750 Km north – south. It is presumed that fires in northern China exhaust an estimated amount of 100 – 200 MT of underground coal which is about 2 – 3 % of world CO_2 production. Up to 10 coal fires per year in the Ruhr area of Germany are caused by spontaneous heating. In it is showed in surveys that 45 of the county's 153 collieries were on fire in 1931 in the West Riding of Yorkshire (England). A more recent example is the spontaneous combustion of ball up heaps at Middleburg colliery in Witbank coalfield in South Africa.

China

In China, coal fires are a severe problem where it is the world's largest coal producer with an annual output around 2.5 billion tons. It has been estimated that some 10-200 million tons of coal are uselessly burn annually, and that the same amount again is made unprocurable to mining. Coal fires extend over a belt across the entire north China, whereby over one hundred major fire areas are listed, each of which incorporates many individual fire zones. They are concentrated in the states of Xinjiang, Inner Mongolia and Ningxia. Beside losses from burned and inaccessible coal, these fires lead to air pollution and substantially increased levels green house gas emanations and have thereby become a problem which has gained international attention. But at the same time, some of the most intensifier fire fighting actions worldwide are being undertaken in China.

Germany

In Planitz, now a part of the city of Zwickau, a coal seam that had been burning since 1476 could only be quelled in 1860. In Dudweiler (Saarland) a coal seam fire erupted around 1668 and is still burning today. Also well-known is the so-called Stinksteinwand (stinking stone wall) in Schwalbenthal on the eastern slope of the Hoher Meibner, where various seams caught fire centuries ago after lignite coal mining terminated; combustion gas continues to reach the surface in these days.

Indonesia

In Indonesia no exact count of coal seam fires has been completed. Coal and peat fires in Indonesia are often ignited by forest fires near outcrop deposits at the surface. Only a small

fraction of the country has been appraised for coal fires. The best data available come from a taxonomic study, on-the-ground observation. A total of 125 coal fires were directed and mapped within a 2-kilometer strip either side of a 100-kilometer elongate of road north of Balikpapan to Samarinda in East Kalimantan, using hand-held Global Positioning System (GPS) equipment In 1998. In the same year extrapolating this data to areas on Kalimantan and Sumatera underlain by known coal deposits, it was estimated that more than 250,000 coal seam fires may have been burning in Indonesia.

United States

In the USA many coalfields are subject to spontaneous combustion. The Federal Office of Surface Mining (OSM) maintains a database (AMLIS), 150 fire zones listed in 1999. There are 45 fire zones are known In Pennsylvania, the most well known being the fire in the Centralia mine in the hard coal region of Columbia County. Coal fires have developed as a effect of variations in the groundwater level, which can increase the temperature of the coal up to 30 ^{0}C, enough to cause it to spontaneously ignite in Colorado. The Powder River Basin in Wyoming and Montana contains some 800 billion tons of brown coal.

Indian scenario

History of coal mines fires can be traced back to the year 1865, when the first fire was reported in Raniganj Coalfields. Mine fires in coal mines in India have survived since the second and third decade of 20th century. However, the accurate history of the first happening of coal mine fires in India has not been accurately recorded. Fires in coal mines are one of the great problems of Indian coal mining industry. In India every year many incidence of the fire come about in the under ground and surface due to spontaneous combustion. Analysis of the reason of the coal mines fire reveal that they start either from an open fire over the external mining means or originate due to varying nature of coal. The chances fires occur whenever and wherever combustible material is present in mine working. Over 140 years fires have been reported till the year1967 from both Jharia and Raniganj coal fields and good quality non-cocking coal in Raniganj coal fields. In Jharia coalfield the first fire occurred in 1916 at Bhowra Colliery. In Indian coal mines 75% of the coal fires take place due to spontaneous combustion. The main face of initiating the fire in India is that the coal seams are thicker and there is a chances of spontaneous heating during the depillaring operation. Coal Mining in country was started in 1774 in Ranigunj Coalfield (RCF) in West Bengal.

Plate 2.1 Coal mines in India

It was started in Jharia Coalfield (JCF) in the last decennium of the 19th century. In the independent India mine fires were known to have existed in the coal mines in Margarita in Assam, Venkatesh Khani Mine in Singareni in Andhra Pradesh, Mahakali Colliery in Chandrapura in Maharashtra, Talcher Colliery in Talcher Coalfield in Orissa, and some mines in RCF in West Bengal and JCF in Jharkhand, etc.In 1966 there were 64 coal mine fires out of which 32 were in JCF and 19 in RCF. The number increased to 82 in 1976. The number of the fires increased further and was about 196 covering an area of about 30 sq km in 1994. The uttermost number of fires, over 70, were in JCF. All the coalfields have mine fires and a sizable quantity of coal has been burnt by the fires. It is not executable to extract all the coal by caving method or even by complete packing under Indian mining condition. Pillars standing for long time are liable to degenerate in straight and spilling may occur. The problem of extraction of thick seam and coal standing in pillars is a severe one particularly in cases where they are with high moisture, high volatile and low ash content which are more nonimmune to spontaneous combustion.

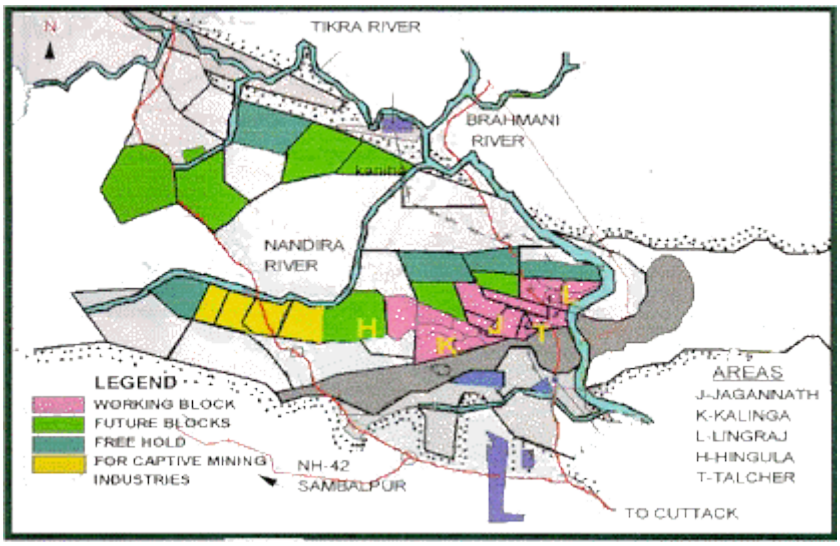


Plate 2.2 Area map of coal fields of MCL

2.3 CONCEPT OF SPONTANEOUS HEATING

Spontaneous heating is defined as "self heating of coal ensuing in its ignition without the application of external heat". Coal interacts with oxygen of air at ambient temperature releasing heat: if allowed to collect would enhance the rate of oxidation and ultimately leads to fire. It is facilitated in circumstances, where large mass of coal is involved and ventilation is either too little to restrict coal-oxygen interaction nor too high to spread out the generated heat. Under these condition, a part of coal mass may heat up to the stage of ignition after the lapse of sure time (known as incubation period). This spontaneous heating is came across by various seam, geological and mining factors.

2.4 MECHANISM OF SPONTANOEUS HEATING

Erdogan Kaymakci and Vedat Didari observed the relation between coal belongingses and spontaneous heating parameters. The oxidation of coal, like all oxidation reactions, is exothermic in character. The accurate mechanism of the reaction is still not well understood. However, scientists agree that the nature of the interaction between coal and oxygen at much low temperatures is amply physical (adsorption) and changes into a chemisorption form commencing from an ambient temperature (Banerjee, 1985). When coal is exposed to air it occupies oxygen at the exposed surface. Some fraction of the exposed coal substance absorbs oxygen at a firmer rate than others and the oxidation results in the establishment of gases. Mainly CO, CO2, water vapor along with the development of heat during the chemical reaction. The rate of oxygen ingestion is extremely high during the first few days

(particularly the first few hours) following the vulnerability of a fresh coal surface to the atmosphere. It then decreases very tardily without causing problems unless rendered heat is allowed to gather in the environment. Under certain statuses, the accumulation of heat cannot be precluded, and with sufficient oxygen (air) supply, the operation may reach higher stages. The CO, CO_2 and H_2O molecules are disintegrated above 70-85^0 C at initial stage to form the loose coal oxygen water complex.

The rate of chemical reactions and exo-thermicity change with the rise in temperature, and radical changes take place, starting at about 100^0 C, mainly due to loss of moisture (Banerjee, 1985;). This process continues with the ascend in temperature, yielding more stable coal-oxygen complexes until the critical temperature is reached. The ignition temperature of bituminous coal is nearly 200^0 C and of anthracite coal nearly 398^0 C. A good air current will effectively prevent oxidation. Once the coal reaches it ignition point, the air supply to it will only increase the combustion. The term spontaneous combustion express a relative value which are separated as highly susceptible or not.

2.5 FACTORS AFFECTING SPONTANEOUS HEATING OF COAL

The main reason for the difficulties in understanding the mechanism of spontaneous combustion is the presence of many internal and external factors affecting the initiation and development of the phenomenon (Table 1). These factors have been reviewed by various researchers (Kr¨oger and Beier, 1962; Guney, 1968; Chamberlain and Hall, 1973a; Feng *et al*., 1973; Beier, 1973; Kim, 1977; Banerjee, 1982; Didari, 1988; Goodarzi and Gentzis, 1991; Didari and ¨Okten, 1994). The main factors which have substantial effects on the process are summarized below:

Table 2.1 Factors affecting spontaneous combustion of coal (Guney, 1968).

Intrinsic Factors (Nature of Coal)	Extrinsic Factors (Atmospheric, Geological and Mining Conditions)
• Pyrites • Moisture • Particle size and surface area • Rank and petrographic • constituents • Chemical constituents • Mineral matter	• Temperature • Moisture • Barometric pressure • Oxygen concentration • Bacteria • Coal seam and surrounding strata • Method of working

	• Ventilation system and air flow rate • Timbering • Roadways

- Pyrite content may speed up spontaneous combustion,
- Changes in moisture content; i.e., the drying or wetting of coal, have evident effects,
- As the particle size decreases and the exposed,
- surface area increases, the propensity of coal towards spontaneous combustion increases,
- It is widely recognized that lower rank coals are more susceptible to spontaneous combustion than higher rank coals. The abnormalcies in this relationship may be assigned to the petrographic components of coal. However, this phenomenon has not yet been fully understood and needs further study,
- Ash content generally decreases the liability of coal to spontaneous heating. Certain parts of the ash, such as lime, soda and iron compounds,
- May have an accelerating consequence, while others, such as alumina and silica, produce a retarding effect. It is clear that some chemicals encourage combustion while others inhibit its development. Also, it is known that oil shale bands abutting coal seams play an important role in mine fires,
- The temperature of the underground atmosphere is a direct component,
- The presence of faults and zones of impuissance around faults may contribute to the danger by permitting air leakage into coal mass,
- Mining methods with overtone extraction, in which part of the coal seam is left in the goaf and pillars (designed for several purposes), can contribute to the possibility for spontaneous combustion,
- Air flow rate is a complex factor because an air supply provides oxygen while it contains away the heat produced. There is a critical air quantity which allows the coal to oxidize and also allow the generated heat to accumulate. Therefore, it prefers the process,
- High ventilation differentials and changes in the mine ventilation system also pretend the development of the spontaneous combustion process.

Table. 2.2 Set elements of mining conditions (Banerjee ,1985)

Sl. No.	Mining parameter conditions	Set elements	Probability of spontaneous fire risk	
			High	Low
1.	Category of coal (Chemical nature)	a) Highly susceptible b) Poorly susceptible	High -	- Low
2.	Friability of coal	a) Highly friable b) Poor friability	High -	- Low
3.	Method of working	a) Bord and Pillar b) Longwall	High -	- Low
4.	State of stowing	a) Extraction with caving b) With complete stowing	High -	- Low
5.	Seam thickness	a) High (>5m) b) Low (<4m)	High -	- Low
6.	State of extraction	a) Partial extraction b) Complete extraction	High -	- Low
7.	Nature of extraction	a) Extraction with more than one slice b) In one slice	High -	- Low
8.	Geological disturbances	a) Present b) Absent	High -	- Low
9.	Rock bumps	a) Present b) Absent	High -	- Low
10.	Dykes	a) Present	High	-

		b) Absent	-	Low
11.	Overburden	a) Greater than 300m b) Less than 300m	High -	- Low
12.	Parting	a) Shale structure b) Rocky and consolidated	High -	- Low
13.	State of consolidation of barrier	a) Fractured and crushed b) Well consolidated	High -	- Low
14.	Scope of accumulation of fines	a) Fine accumulation sustained b) Fines avoided	High -	- Low
15.	Method of ventilation	a) Advancing type b) Retreating type	High -	- Low
16.	Quantity of ventilation	a) Intensity of pressure difference high b) Low pressure difference	High -	- Low
17.	Humidity	a) Wet mines b) Dry	High -	- Low
18.	Source of hot spots	a) Present b) Absent	High -	- Low
19.	Gas emission rate	a) Low b) High	High -	- Low
20.	Size of panel of the face	a) Large b) Small	High -	- Low
21.	Rate of face advance	a) Slow	High	-

		b) Fast	-	Low
22.	Chances for blockage of face advance	a) Present b) Absent	High -	- Low

2.5 THEORIES OF SPONTANEOUS COMBUSTION OF COAL

2.5 .1 Pyrite theory (Banerjee et.al., 1985)

In pyrite mines Heating due to oxidation of pyrites are very common process.

Heating of coal could be caused by iron pyrites and in finely powdered and dispersed state in the presence of moisture. The reaction of iron pyrites with oxygen and moisture gives products of larger volume than the original pyrite hence opening more pore area of exposure for oxygen, which is a exothermic reaction.

$2Fe S2+ 702+ 16H2O = 2 H2SO4+ 2 Fe S04.7 H2O + 316$ kcal

The above equations show towards an exothermic reaction. It also yields reaction product having greater volume than the actual pyrite, with the result that would break open any coal in which they are engrafted and thud exposing a greater surface of coal to the air. It is also observed that sulphonated coal have greater reactivity towards oxygen, especially in the presence of iron oxide. It might have an considerable effect if its concentration in finely dispersed from exceed 5 to 10 %. If the pyrite present is less than 5%, then its effect is negligible.

2.5.2 Bacterial Theory (Banerjee et.al., 1985)

Bacteria were also encourage self- ignition of coal. The contribution of heating due to the action of bacteria could not be rejected. It is not also entirely successful due to the action of bacteria. The activity of bacterial are main cause of Spontaneous heating which observed in haystacks and in wood.

However, there is no conclusive proof to authenticate or dispose this theory. Hence it is concluded that bacteria could cause only a slight heating which may not play any important role.

2.5.3 Phenol Theory (Jain, 2009)

theory is concerning because it offers a method of determining liability of coal to spontaneous heating. There are many Experiments have shown the result that phenolic hydroxyls and poly phenols oxidize faster than other groups.

2.5.4 Electro-chemical Theory (Jain, 2009)

The electrochemical theory also on factor which could cause spontaneous heating of coal. It describes auto-oxidation of coals as oxidation-reduction processes in micro galvanic cells made by the coal components.

2.5.5 Coal oxidation theory (Banerjee et.al., 1985)

The establishment of peroxyl radical and hydro peroxides is commonly to be conceived to be they mechanism by which oxygen and moisture are initially in corporate into organic matrix. These species may rearrange, react or decomposed to form wide range of oxygen functionality in the gaseous product

The following factors are favourable for The oxidation process of coal:

- **Type of coal** – Usually high moisture and low rank coal are higher oxygen avidity with better formation of peroxy-complex, hence higher tendency towards spontaneous heating. it is the accessibility of active canters in coal that defines the proneness of a definite coal to spontaneous heating as the intrinsic oxidation mechanism is same for every type of coal.
- **Extent of oxidation** – As the time of exposure of coal surface to air gains the oxidation reaction gradually decreases and the coal gets oxidised. In the initial stage the macro pores on surface determine oxygen consumption whereas in later stages, it finds out the micro pores.
- **Temperature** – The lower limit temperature for coal-oxygen reaction is -800 ^{0}C during which it is physical adsorption and at room temperatures it is chemisorption. Usually rate of chemical reaction increases with temperature develop and is almost doubled for 100 ^{0}C rise in temperature.
- **Moisture** – Moisture helps in the formation of proxy complex and influences the rate of reaction. Release of moisture from coal produces more active centres making it more powerful to oxidation. It adds to heat need for spontaneous combustion by heat released.

Various stages of coal oxidation is given in the flow chart below :

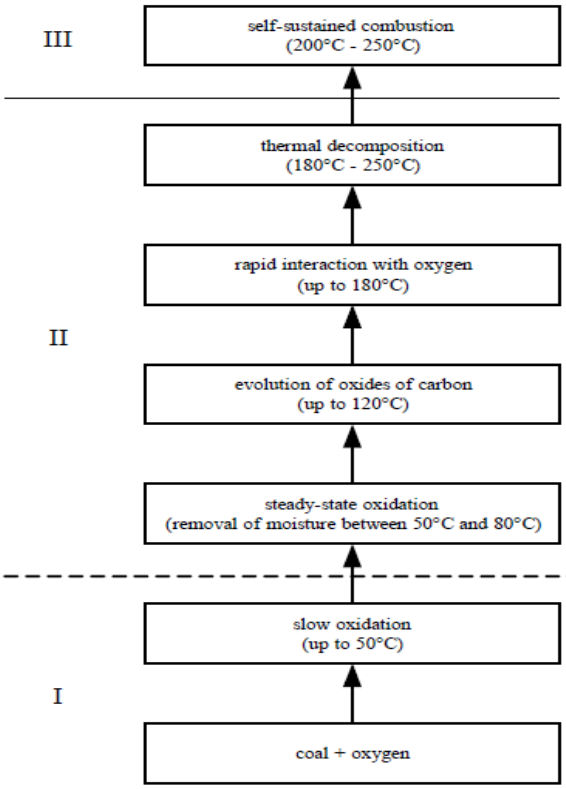


Fig 2.1 Sequential stages in the spontaneous combustion of coal (Banerjee et.al., 1985)

2.5.6 Humidity theory (Jain, 2009)

Humidity theory states, the quantity of heat released by atmospheric oxidation of coal is much less than the amount of heat required removing water from the coal. If the evaporation of water can be caused at the seat of heating, then the temperature of heating would decrease. When it is recollected that water is an oxidation product of low temperature oxidation of coal, the above scheme well justifies other possible sources of CO and CO_2 in low temperature reaction between coal and oxygen. For a normal moist coal (65% relative humidity) heat of wetting could cause a temperature rise of only 2° C, which would be unconvincing to contribute significantly to self-heating in store coal. Although dry coal expressed a substantial rise of temperature, heat of wetting (6 KJ / Kg) values decreases quickly with increasing moisture content and heat.

2.6 LITERATURE REVIEW

The following is the brief review of the work carried out by different researchers to determine the spontaneous heating tendency of coal samples.

Banrjee and Chakravarty (1967): Have proposed differential temperature Analysis(DTA) for the study of spontaneous combustion of coal, especially in classifying coals with respects to their susceptibility to self heating. A standard experimental procedure for carrying out DTA studies had been prescribed by them. Calcined alumina is recommended as inert reference material for DTA experiments. A heating rate of 50 ^{0}C/min was proposed for such studies. Typical temperatures found from different coals are includes in this study to explain self heating phenomenon.

Bhattacharya (1971): Expressed out laboratory experiments to evaluate the rates of heat exhaust from different coals by a calorimeter during sorption of water vapour in isothermal conditions. It was found that the rate of heat generation in a particular coal increases with the equilibrium humidity insufficiency of the coal, i.e. with the difference of equilibrium humidity of air and coal. For a given coal, the rate of heat propagation due to oxidation has been found to be negligible in comparison with that due to sorption of water vapour. A small peak‘ at the starting of the rate curves has been observed during the tests with dry coals, with the exclusion of anthracite; accounts for this phenomenon have been assayed. The results also show that under a given test condition the characteristic rate of heat release is dependent on the type of coal, its particle size and its weathering.

Banerjee (1972): Determined the Crossing Point Temperature (CPT) of a number of Indian coal samples adopting the Crossing Point Temperature method. He found that coals with crossing points temperatures above 160 ^{0}C are badly susceptible. Between 120 ^{0}C & 140 ^{0}C could be considered to be extremely susceptible to spontaneous heating.

Nandy et al. (1972) : They noted the fluctuation in Crossing Point Temperature values with the moisture content, oxygen percentage and the volatile matter of coal. He observed that CPT normally minimizes with the maximizes in each of these constituents of coals. But outside 35% V.M, 9% oxygen, or 4 to 6% moisture content there is not much change in CPT values. In fact, above 4 to 6% moisture content in coal, then CPT values shows a developing trend.

Mishra et.al. (1980): Introduced a critical analysis of different genetic and physical factors related with certain Early and Late Permian and Oligocene coal seams and early Eocene lignite seams of India disclosed that the high releasing porosity and small particle size, irrespective of petrographic and rank properties in sub-humid climate, favour spontaneous combustion. Large particle size, high rates of gas expelling and extremely wet or dry seams, in combination with each other tend to inhibit spontaneous heating in sub-humid, per-humid and semi-arid conditions. The susceptibility to auto-ignition of a coal or lignite seam is not

uniform during its lateral extent are not sufficient to cause auto-ignition but due to high integral porosity at any given rank stage and a high content of susceptible or oxidizable components.

Benerjee (1985): He derived the formula for the olpinski index, in this method small pellet of coal is allowed to indergo aerial oxidation at temperature around 135 ^{0}c gives the measure of spontaneous heating susceptibility SZ_a of the concerned coal. Olpinski method makes correction for ash content of the coal ($A_{a)}$, and expressed spontaneous heating index as:

$$SZ_b = SZ_a - 100/ (100-A_a)$$

Tarafdar et.al. (1987): They reported results of wet oxidation of coal using alkaline permanganate solution involving measurements of differential temperature at various temperatures, at a changeless heating rate, and possible changes between a saturated calomel electrode and a carbon electrode immersed in the coal oxidant mixture within a definite reaction time at a invariant temperature. The measurements is being made on seven coal samples of different coalfield of known crossing point temperatures (CPT). 4 samples, conceived to be highly susceptible to spontaneous heating, had CPT in the range 132-137 ^{0}C, and three, considered badly susceptible to spontaneous heating, had CPT values in the range 162-1680C, showing two different zones of correlation between CPT values and the agreeing differential peak temperatures, and between CPT and the found potential changes. It was proposed that differential temperature and potential difference measurements at the time of wet oxidation of coal may be used as alternative techniques for the assessment of propensity to spontaneous heating.

(Karmakar et.al , (1989): They observed the three methods for determining spontaneous combustion susceptibility of cola have been sorted out the experimental data obtained by these methods for sixteen coal samples. This study brings out that a linear relationship exist amongst CPT, polish SZ and Russian U indices. Coal with low moisture and high VM content being high susceptibility to spontaneous combustion. SZ_a index being a convenient and quick method can be applied as an substitute to CPT method now being complied in India. In this method a small pellet of coal is appropriated to undergo aerial oxidation at a temperature about 230 ^{0}c with the help of quinoline liquid.

Tarafdar and Guha (1989): They conveyed out a overture investigation. using wet oxidation method , and they proposed that a systematic and thorough study along this line is needed as the significance of this technique.

Banerjee et. al., (1989): Studied the advantages and disadvantages of different fire indices in common use, such as CO/O_2 deficiency (def.) %, CO_2/O_2 def % and CO/CO_2 percentage, as

well as those proposed in the literature such as the disappeared hydrocarbon index, the CO residual gas relationship and the C/H ratio of the products of combustion, are discoursed in terms of their applicability for finding the extent and intensity of underground sealed fires. Five cases of different types of fires were studied and verified using the above indices. The depiction of fires should be made from fire indices, such as the C/H ratio, CO/O_2 defficiency percentage and the CO/CO_2 percentage, temperature profile studies, pressure differential measurements in stopes and spontaneous heating characteristics of the coal concerned in relation and that inferred from other mining factors. The initiation of active fires are declarative upon the values of C/H ratio above 3 or 4, values of the CO/O2 defficiency percentage of > 0.5 and values of CO/CO2 of > 2. A C/H ratio of 20 shows a dazzling fire where the possibilities burning of wooden props.

Chandra et.al. (1990): As the coalification increases, the intensity of spontaneous combustibility decreases step by step from highly susceptible to moderately susceptible to least susceptible to the spontaneous combustion stage. Proceeded a preliminary survey of the frequency of happening of fire due to spontaneous combustion in the different seams of the Raniganj Coalfield and directed the 14 possibility of a relationship between coalification and spontaneous combustion of coals. Besides rank, as proved from reflectance studies, the amount of vitrinite and exinite contents of the coal seams also determined the spontaneous combustibility of the coal seams. They observed that pyrite of the Raniganj Coalfield had no influence on the combustibility of the coal seams. It was resolved that the proneness to spontaneous combustion of the coals is concerned to coalification.

Gouws, et al. (1990): Designed an adiabatic calorimeter to enable the spontaneous combustion propensity of coal to be based. Various indicators of self-heating potential, such as total temperature rise, initial rate of heating, lower limit self-heating temperature, and kinetic constants were inquired. Results experienced from the adiabatic tests were compared with the results of crossing-point temperature determinations and differential thermal analysis (DTA) tests for the same coals, with a view to developing a mathematically consistent spontaneous combustion liability index. This paper draws the major components of the adiabatic calorimeter.

Jose et .al., (1995): As the rank of the coal increases, both the self-heating and the end of combustion temperatures also increase. The total heat loss (area under the DTA curve) increases with the rank of the coal. They applied differential thermal analysis (DTA) as a method to study the self-heating behaviour of fresh and oxidized coals. Oxidation was executed in air at 200 °C for periods of up to 72 hour. An increase in the self-heating

temperature, a decrease in the temperature of the end of combustion and a decrease in total heat flow were found as a consequence of coal oxidation. A relationship between the total heat loss and the calorific value as found out using the ASTM standard method is pointed out.

Panigrahi et.al., (1997): It has also been observed that from point of susceptibility of spontaneous heating, Russian index shows similar relation with the basic components of as the crossing point temperature, which may demonstrate to the handy method of coal classification in Indian context. conducted experiments for the determination of Russian U-index, 10 samples from Jharia coalfields have been verified using this method. The crossing point temperature of these samples have also been determined carbon, hydrogen, nitrogen and sulphur contents for this samples have been determined by using Fenton's method of ultimate analysis, in addition. Then efforts have been made to correlate the Russian index and CPT of coal samples with its basic constituents viz. carbon, hydrogen and ash contents.

Tripathy et.al., (2001): They found out the different parameters of spontaneous heating as well as the different followed commonly in india. In this paper hey specify the mechanism of spontaneous heating & different indicies used by different researchers.

Kaymakci et.al.,(2002): According to the multiple regression analyses, these major factors are volatile matter, carbon, hydrogen, nitrogen (N), oxygen (O), sulphur (S) and inertinite. They also carried out linear and multiple regression analysis to determine the relationship between spontaneous combustion parameters and coal parameters have been formulated. They have derived some empirical equations using statistical models. The linear regression analysis have shown that ash (A), volatile matter (VM), carbon (C), hydrogen (H), exinite (E), inertinite (I) and mineral matter (MM) are the major components affecting spontaneous combustion.

Panigrahi et.al. (2004): Carried out extensive field in India, studies to inquire the pillar fire problems in one coal mine. A Thermal IR gun and a Thermo vision camera have been employed for thermal scanning to evaluate the state of heating in chosen pillars. Special sampling setups have been planned to collect gas samples from the holes drilled into the selected pillars and the multi gas detector is used to examine the composition of samples in situ. These ratios may also be used for assessing the condition of fires in sealed-off areas.

In order to predict the spontaneous heating in coal pillars, different gas ratios have been computed and it has been observed that some of the founded gas ratios, such as; Graham's ratio, Young's ratio etc. have resulted in negative values in samples holding high amounts of methane. The modified gas ratios have been suggested which will be useful for predicting the pillar fires.

Singh, et al. (2007) : The problem of spontaneous heating is very common in coal mines all over the world. The coal industry faces many difficulties day by day due to the occurrence of spontaneous heating in the open pit coal mines. Presently, about 70 per cent of total coal production in Indian mines is being accomplished by open pit mining. Fire in coal, particularly in open pit mines not only causes reparable loss of natural wealth but also damages the surface structures and pollutes the environment. It is well known that 75 per cent of the fires occur due to the spontaneous combustion of coal. They found in opencast mines, coal immediately oxidises and catches fire due to the intrinsic features of coal, In opencast mines, when the coal benches are left idle for a longer time, heat accumulation takes place in prosperous conditions and sometimes leads to fire, such as low rank, high moisture, high volatile matter, presence of sulphur in the form of pyrites, low crossing point temperature (CPT) and ignition point temperature (IPT) value and less incubation period. The intention of this paper is to present the different successful case studies concerning the safety management of open pit coal mines from occurrences of spontaneous heating.

Ahmed, et al. (2008): They Calculated Liability index using CPT for studying the propensity of coal towards spontaneous heating. CPT that has been in lab needs extra precaution for repeatable results. Among these, crossing point temperature (CPT) method is very simple and gives a good idea of the propensity of coal towards heating. Liability index calculated using CPT is also useful for studying the propensity of coal towards spontaneous heating. Also the correlation between liability index and crossing point temperature have been presented in this paper. Many attempts were taken to study the relationship between peripheral oxygen groups or the functional oxygen groups in coals and their correlation with proneness to auto-oxidation using liability index (LI) to overcome the difficulties.

Sahay et.al., (2008): They Proposed characterisation of coal towards spontaneous heating for taking corrective measures realistically. Based on thermal study, the correlation with liability index model established on coal proximate analysis results of coal sample including surface area and porosity. They proposed a brief description of different liability index particularly a critical analysis of dependency of critical temperature on moisture content, ash content, volatile matter, carbon content, surface area and porosity, new liability index based on thermo decompositional study of coal sample. They formulated a methodology based on thermo-decompositional study of coal sample for finding of minimum temperature at which coal bed temperature starts self propulsive known as critical temperature and a new liability index based on it.

Mohalik, et al., (2009) : In this year they presented the review of application of three thermal techniques such as differential thermal analysis (DTA), differential scanning calorimetry (DSC) and thermogravimetry (TG); for studying the susceptibility of coal to spontaneous heating and fire.

They also critically analyses the experimental standards take by different researchers, while applying these techniques in studying thermal behaviour of coal samples. The paper also delivers the future guidance of research in this subject area.

Nimaje et.al. (2010): They made a view on thermal studies carried out by different researchers across the globe for determination of spontaneous heating of coal and revealed that lot of emphasis on experimental techniques is necessary for evolving appropriate strategies and effective plans in advance to prevent occurrence , ignition and spread of fire. They also studied on thermal studies on spontaneous heating of coal of all the experimental techniques developed thermal studies play an important and dominant role in assessing the spontaneous heating susceptibility of coal.

CHAPTER – 3

EXPERIMENTAL TECHNIQUES

The study of various parameters of coal that affect the spontaneous heating tendency of coal, the following experiments are needed to be carried out:

1. Proximate analysis
2. Ultimate analysis
3. Calorific value
4. Flammability temperature
5. Wet oxidation potential
6. Crossing point temperature
7. Olpinski index
8. Russian U- Index Method
9. Differential thermal analysis
10. Differential scanning calorimetry

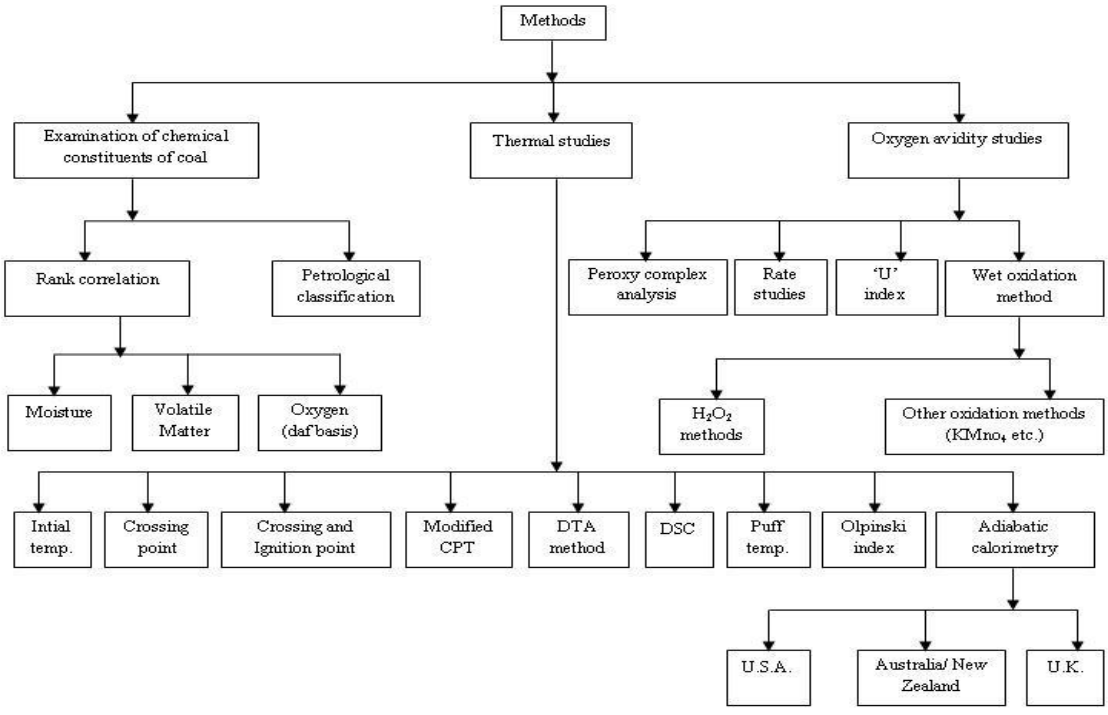


Fig 3.1 Methods of spontaneous heating (Banerjee et.al., 1985)

3.1 SAMPLE COLLECTION AND PREPARATION

Sampling is the process of collecting a small portion of a whole such that the substance of that constituent represents that of the whole. It is the process by which physical and chemical features of a mineral or ore are determined with the desired accuracy. In the case for coal it covers the properties ascertained by proximate and ultimate analysis such as fixed carbon, volatile matter, ash, caking index and calorific value etc. the physical nature of the ore is sometimes requirement to be determined and the sampling process assumed should be able to give this information too. Samples are generally collected at regular interval. The interval of sampling point is governed by the regularity of the deposits as well as the accuracy for sampling projected. It should be coned and quartered on the sampling sheet, if the quantity is large before collecting it sampling bags. There is always change that missing of collected samples whereas the reduction in volume of samples has to be done individually. Different types of sampling are:

a) Bulk sampling
b) Chip sampling
c) Drill hole sampling
d) Grab sampling
e) Channel sampling

Bulk sampling is the method of sampling and it is done where formal sampling methods do not give a instance scale, bulk sampling resorted to or large scale sampling. Bulk samples excrete the effect of irregular distribution of value.

Chip sampling can be taken in case of uniform ores and where the rock structures are independent of the values. It is done in hard ores where it is hard to cut the channels. It The sample is collected by breaking of small same sized chips from a face at points usually as spaced both horizontally and vertically.

Grab sampling is usually grab sampling of the ore broken in the stope is unreliable as accurate estimation of the volume of broken ore is unacceptable. In the stope or at the face which carried ore where the broken ore are applied,. Grab sampling of tubs or ships are collected from units of regular volume, are however more representations since samples.

For the project, the samples were collected by Channel sampling method, which is the most common method followed throughout in sample collection of various mines.

3.1.1 Channel sampling (IS 436 Part I/Section I - 1964)

In channel sampling the section of seam to be sampled shall be exhibited from the roof to the floor. The particular seam sample shall, be taken in a channel representing the total cross-section of the seam having the dimensions of 30 x 10 cm, i.e. 30 cm in width and 10 cm in depth. For the channel sampling purpose there are two parallel lines, which are 30 cm obscure end at right angles to the bedding planes of the seam shall be labeled by a chalked string on the plane, freshly exposed surface of the seam. It is Obvious that, dirt bands exceeding 10 cm in thickness shall be omitted. The channel between the marked chalk lines in the seam shall be cut to a depth of 10 cm and the coal sample collected on a clean strong cloth or tarpaulin placed instantly at the bottom so that the chances of pieces flying off during excavation of coal are understated.

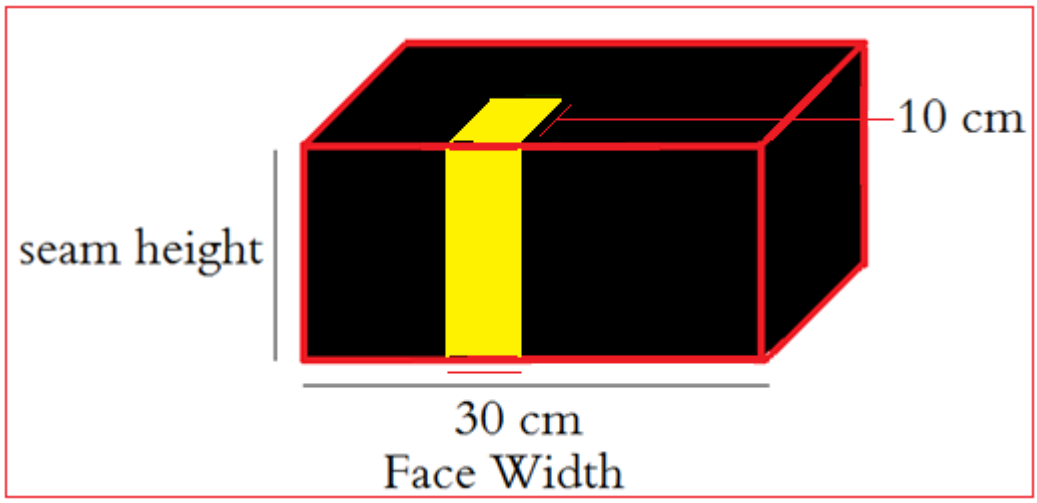


Fig 3.2 Channel Sampling

3.1.2 Sample preparation (IS 436 Part-I/Section 1-1964 and IS 436 Part II-1965)

The samples are taken from the different coalfield by channel sampling are broken in crusher or as per the experimental method necessities in the laboratory. The crushed sample are then sieved by the different sieve to required sizes and stored in air tight polythene packets. The packets are then stored in air tight containers for further use in the experiments.

3.2 METHODS FOR DETERMINING INTRINSIC PROPORTIES OF COAL

3.2.1 PROXIMATE ANALYSIS (IS 1350 Part I -1984)

Proximate analysis was developed as a simple mean of determining the distribution of products found of coal. When the coal sample is heated under specified conditions, then it

classifies the products into four groups: i) moisture; ii) volatile matter iii) fixed carbon, iv) ash, the inorganic residue remaining after combustion. For proximate analysis, i.e. for the determination of volatile matter, moisture, ash and fixed carbon, the method determined by IS (Indian standard) 1350 (Part-I) – 1984 was followed.

3.2.1.1 Determination of moisture content (M)

Coal is always associated with some amount of moisture, which is both physically and chemically bound , due to its nature, origin and occurrence. It is customary to differentiate between extraneous and inherent moisture. When a wet coal is exhibited to atmosphere, the external moisture evaporates, but the obviously dry coal still contains some moisture, which can be removed only on heating above 100 ^{0}C. External moisture is also called accidental or free moisture, whereas inherent moisture is named as equilibrium or air-dried or hygroscopic moisture. The quantity of external moisture counts mainly on the mode of occurrence and handling of coal, but the air-dried moisture is associated to the inherent hygroscopic nature of the coal.

Experimental Procedure :

- About 1g of finely pulverized -212 micron (BSS) air-dried coal sample is weighed in a silica crucible and then placed within an electric hot air oven.
- It is maintained at 110^{0}C.
- The crucible with the coal sample is allowed to put in the oven for 1.5 hours and it is taken out with the help of tongues, then cooled in a desiccator for about 15 minutes.
- Then weighed. The loss in weight is reported as moisture (on percentage basis).

The calculation is done as per the following.

$$\% \text{ Moisture} = \frac{Y-Z}{Y-X} \times 100$$

Where,

X = weight of empty crucible, in grams (gm)

Y = weight of crucible + coal sample before heating, in grams (gm)

Z = weight of crucible + coal sample after heating, in grams (gm)

Y -X = weight of coal sample, in grams Y- Z = weight of moisture, in grams (gm)

3.2.1.2 Determination of volatile Matter (VM)

When coal is heated in defined equipment under appointed conditions, is concerned to as volatile matter, the loss of mass and corrected for moisture. The matter lost is composed of

materials that form upon the thermal decomposition of the various constituents of coal. Some of the element of coal volatile matter are hydrogen, carbon monoxide, methane and other hydrocarbons, tar vapours, ammonia, some organic sulphur and oxygen containing deepens and some incombustible gases, such as carbon dioxide and water vapour, all of which come from the decomposition of organic materials in coal. Inorganic materials in coal contribute the water of hydration of mineral matter, carbon dioxide from carbonates and hydrogen chloride from inorganic chlorides to the volatile matter.

Experimental Procedure :

- For determining the volatile matter a special volatile matter silica crucible (38mm height, 25mm external diameter and 22mm internal diameter) was used.
- First the empty silica crucible along with the lid uncovered was heated at 800 ^{0}C for an hour in the muffle furnace and then cooled to room temperature.
- The empty volatile matter crucible was then weighed again.
- Approximately 1gram of coal sample was weighed in the volatile matter crucible and it was placed inside the muffle furnace maintained at 925 ^{0}C with the lid covering the crucible.
- The heating was carried out exactly for 7 minutes, after which the crucible was removed, cooled in air and then in a desiccator and weighed again.

$$\% \text{ Volatile matter} = \frac{Y-Z}{Y-X} \times 100 - M\%$$

Where X = weight of empty crucible, in grams (gm)

Y = weight of crucible + coal sample before heating, in grams (gm)

Z = weight of crucible + coal sample after heating, in grams (gm)

Y -X = weight of coal sample, in grams (gm)

Y- Z = weight of volatile matter + moisture, in grams (gm)

3.2.1.3 Determination of Ash (A)

During the ashing process, the coal ash is the residue left after the combustion of coal under defined conditions. It does not occur as such in the coal, but is formed as the result of chemical changes that take place in the mineral matter. Ash and mineral matter of coal are therefore not identical The extraneous and inherent mineral matters are the two types of ash forming materials in coal. The extraneous mineral matter consists of materials like calcium, magnesium and ferrous carbonates, pyrite, marcasite, clays, shales, sand and gypsum. The

extraneous mineral matter owes its origin to i) The substances which got linked with the decaying vegetable material during its transition to coal, which is difficult to remove by mechanical methods, and ii) Rocks and dirt getting mixed up during mining and handling of coal. The representation of inherent mineral matter the inorganic elements combined with organic components of coal. The origin of such materials is likely the plant materials from which the coal is formed. Ash from inherent mineral matter is unimportant as far as the total quantity of ash is pertained. But Indian coals suffer from the major disadvantage, that the mineral matter content is not only high, but of intimately associated type, due to its drift origin. The several changes that occur, such as loss of water from silicate minerals, loss of carbon dioxide from carbonate minerals, oxidation of iron pyrite to iron oxide, and fixation of oxides of sulphur by bases such as calcium and magnesium. Because ash is quantitatively and qualitatively different from the mineral matter originally present in coal. In fact, incineration conditions determine the extent to which the weight change takes place and it is essential that standardized operations should be closely followed to ensure reproducibility.

Experimental Procedure :

- weight of the empty crucible is taken.
- 1gm of desired coal sample is weighed in the crucible and is taken in a muffle furnace at 450 ^{0}C for 30 minutes and the temperature of the furnace is raised to 850 ^{0}C for 60 minutes.
- After that time interval, the crucible is taken out and placed in a desicator and weighed.

$$\% \text{ Ash} = \frac{Z-X}{Y-X} \times 100$$

Where

X= weight of empty crucible in grams (gm)

Y= weight of coal sample + crucible in grams (gm) Before heating

Z= weight of coal sample + crucible in grams (gm) After heating

3.2.1.4 Determination of Fixed Carbon (FC)

The mathematical remaining after the determination of moisture, volatile matter and ash is fixed carbon by defination. In fact a measure of the solid combustible material in coal after the expulsion of volatile matter. Fixed carbon plus ash present the approximate yield of coke from coal. The value of fixed carbon is determined by subtracting from 100 the resultant

summation of moisture (M), volatile matter (VM) and ash (A). with all portion on the same moisture reference basis.

$$FC = 100 - (M+MV+A)$$

Where,

moisture (M), volatile matter (VM), Ash (A) content of coal.

3.2.2 ULTIMATE ANALYSIS (IS 1351 - 1959)

The objective of coal ultimate analysis is to determine the basic chemical constituents present in the coal. The ultimate analysis determines the amount of carbon (C), hydrogen (H), oxygen (O), sulfur (S), and other elements among the coal sample.

3.2.3 CALORIFIC VALUE (IS 1350-1959)

The calorific value of coal is the amount of potential energy in coal that can be changed into actual heating ability. The value can be estimated and compared with different grades of coal or even other materials. Materials of different grades will produce differing quantities of heat for a given mass. The calorific value of coal is commonly determined by the bomb calorimeter method. This apparatus consists of a cylindrical chamber known as bomb and it is made up of stainless steel. This chamber is equipped with an air tight cover which can be screwed on the chamber. There are three terminals; two for sparking and one for the entry of oxygen. After forcing the oxygen into the chamber the enactment can be blocked by screwing in the third terminal.

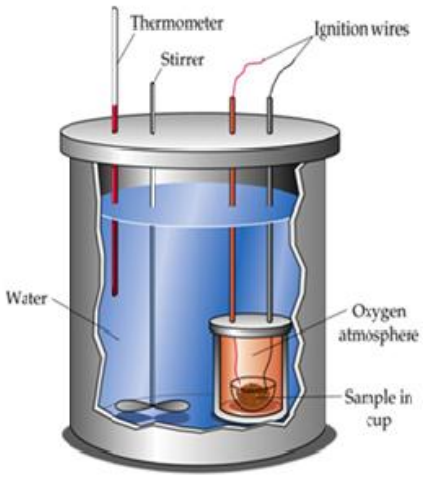


Fig 3.3 Structure of Bomb

On the other side of the cover, there are two bent rods connected to two ends. The bent rods have small holes through which two fuse wires are connected. There is the provision for putting the crucible containing the pellet tied to the fuse wire by means of a cotton thread. This whole set-up is placed in a bigger vessel holding a quantity of water in it. A stirrer is used for stirring the water in the bigger vessel.

Experimental Procedure :

- 1gram (gm) of air dried coal sample is taken by weighing in a balance of -212 size (BSS).
- A pellet is made with the coal and weighed. The calorimeter cover is taken and about 10 ml/min of distilled water is poured into it.
- The pellet in the crucible is brought in contact with the fuse wire by means of a thread. The cover is then tightened.
- Oxygen is then admitted into the calorimeter at a pressure of about 300 - 400 psi atmospheres.
- Then 2L of water is put into the bigger vessel.
- Necessary electrical connections are made and stirrer is adjusted in the adjusted position.
- The stirring is done softly for 5 minutes.
- The initial temperature reading is then taken.
- A sparking and combustion of coal has taken place in the calorimeter after the fire of bomb. The maximum reached temperature is then noticed.
- The bomb is removed and the pressure exhausted.
- The bomb internal is examined for unburnt or sooty deposits. If such material is found, then the test is discarded.

The calorific value of coal is calculated by:

Calorific value = (T2 – T1) * Water equivalent) / Weight of the pellet
Where,
T2 – Final temperature
T1 – Initial temperature
Useful heat value (UHV) = 8900 - 138(A+M)

Table 3.1 Grading of non coking coal (IS 1350 - 1959)

UHV (Kcal/Kg)	A + M % (60% RH and 400 C)	GCV (Kcal/Kg)	Grade
Exceeding 6200	Not exceeding 19.5	Exceeding 6454	A
Exceeding 5600 but not exceeding 6200	19.6 to 23.8	Exceeding 6049 but not exceeding 6454	B
Exceeding 4940 but not exceeding 5600	23.9 to 28.6	Exceeding 5597 but not exceeding. 6049	C
Exceeding 4200 but not exceeding 4940	28.7 to 34.0	Exceeding 5089 but not Exceeding 5597	D
Exceeding 3360 but not exceeding 4200	34.1 to 40.0	Exceeding 4324 but not exceeding 5089	E
Exceeding 2400 but not exceeding 3360	40.1 to 47.0	Exceeding 3865 but not exceeding. 4324	F
Exceeding 1300 but not exceeding 2400	47.1 to 55.0	Exceeding 3113 but not exceeding 3865	G

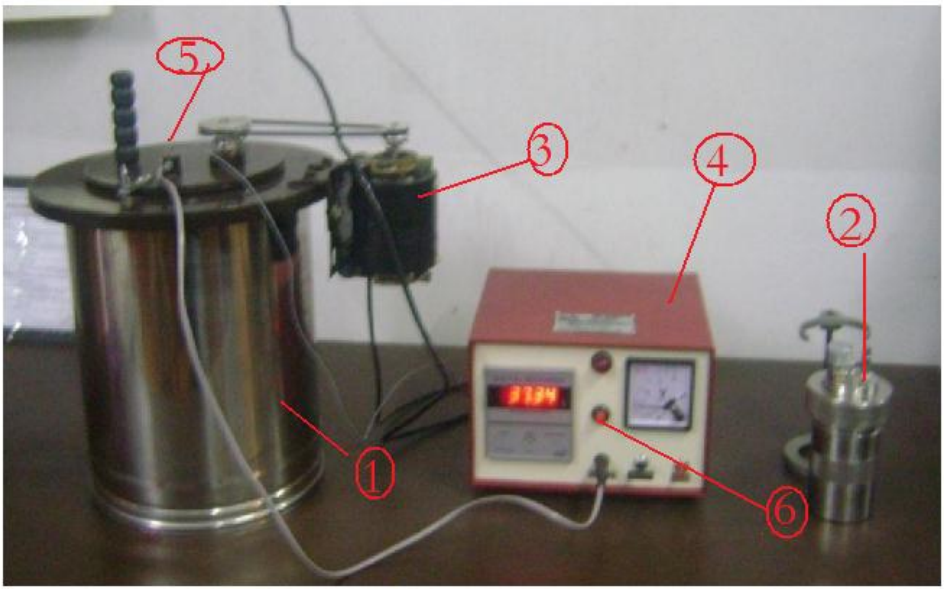


Plate 3.1 Experimental set up of Bomb Calorimeter

1. Calorimeter vessel
2. Bomb
3. Stirrer
4. Read out unit
5. Cover
6. Firing Switch

3.3 METHODS FOR DETERMINING SPONTANEOUS HEATING SUSCPETIBILITY OF COAL

3.3.1 FLAMMABILITY TEMPRETURE METHOD (Nimaje et.al., 2010)

Flammability of explosiveness of the coal dust is defined as its ability. To determine the efficiency of coal dusting, it is essential to carry out the flammability test of coal dust with the help of flammability apparatus. For the finding of the flammability temperature of coal consists of vertical tubular furnace of internal diameter 50mm, length 300mm, open at both ends, a dust dispersing unit, a solenoid valve a reservoir for air, a mercury manometer, a drying tower and an aspirator bulb. Coal dust sample is kept in the helical dust disperser. Air from the reservoir is made to pass through the disperser and on emergency from the divergent mount, forms a uniform dust-air mixture inside the furnace. The lowest temperature at which this mixture catches fire, which is indicated by the appearance of flame coming out of the bottom of the tubular furnace is the flammability temperature of the coal dust.

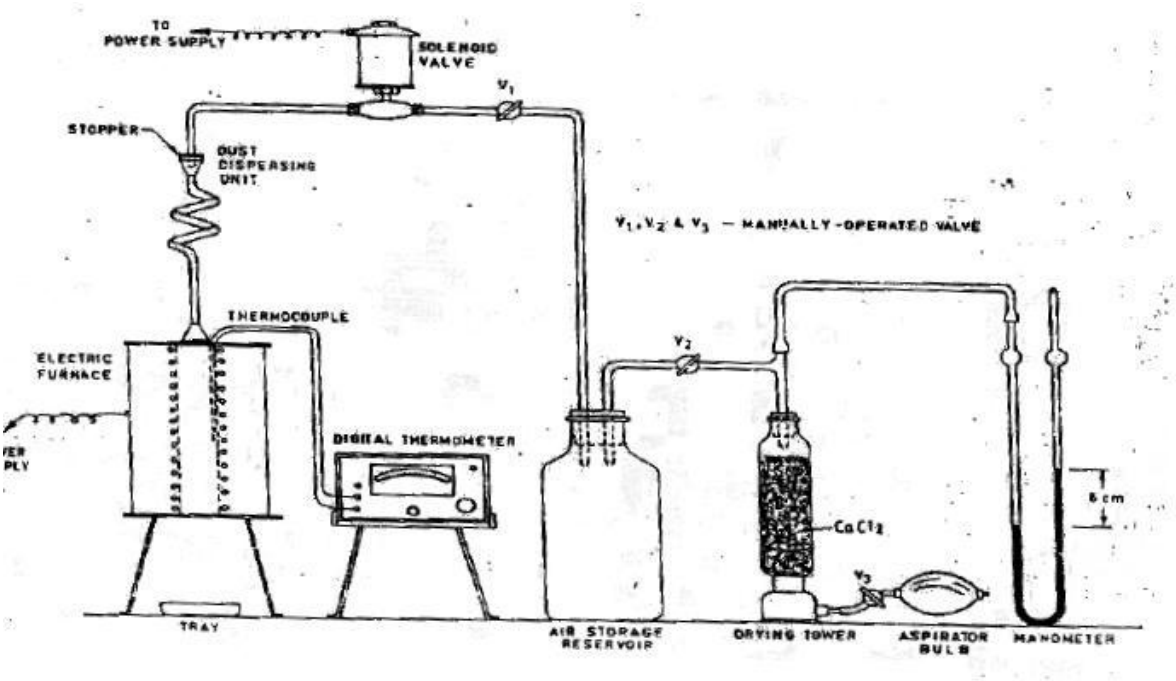


Fig 3.4 Schematic diagram of flammability temperature (Tripathy et.al., 2001)

Experimental Procedure:

- 200 mg sample of coal having mesh size -200 mesh (BSS) Placed in a helical tube.
- By aspirator bulb and turning off the tap the mercury column difference is maintained at of 8 cm.

- After switch on the solenoid valve, at desired temperature of furnace, which allows the air to pass through it very fast and find out the status of coal sample (spark, smoke or flame).
- Incase flame appears then find out the exact temperature in lower temperature range by trial and error method, if it is not appears then go for higher temperature range.

Plate 3.2 Flammability temperature apparatus

3.3.2 WET OXIDATION POTENTIAL (Tarafdar et.al., 1989)

The coal molecule may be considered as consisting of two parts such as one is condensed aromatic structure, which are resistant to oxidation and other one is the aliphatic or hydro aromatic structure that are more prone to oxidation and Presence of hydroxyl group in the aromatic structure part gives a very high degree of responsiveness to coal structure and they get oxidized faster. This is the cause for low rank coals are rather easily oxidized due to the above and also due to smaller degree of contraction of aromatic structures in them. Because low rank coals carry more branched aliphatic hydro-carbons. Since the high rank coals have structure close to that of graphite, it is less liable to oxidation and the products contain more aromatics than aliphatic. Lower rank coals on oxidation method large amounts of aliphatic acids compared to higher rank coals. When coal is contributed to alkaline permanganate, oxidation takes place and the concentration of manganite ion in solution increases relative to permanganate and there will be outcome change in the potential till all the oxidation possible in coal molecule is finish. Therefore, addition of coal to alkaline permanganate solution results in a change of potential of carbon electrode sank in the solution.

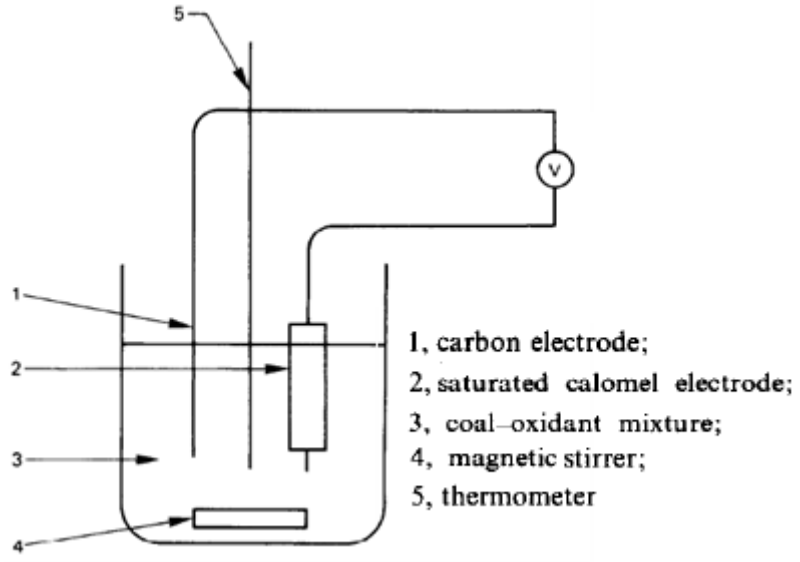


Fig 3.5 Schematic diagram of wet oxidation potential appratus (Tarafdar et.al., 1989)

Experimental Procedure :

- The electrodes is placed In the beaker along with a magnetic stirrer such that a homogeneous mixture of coal and alkali solution is maintained.
- The teflon coated fish of the magnetic stirrer is placed inside the beaker. 0.5 g of coal sample of –212 micron size was mixed with 100 ml of decinormal solution of potassium permanganate (KMnO4) in 1N potassium hydroxide (KOH) solution in a beaker and the coal sample was subjected to wet oxidation process.
- The suspension of coal-oxidant was continuously stirred by using the magnetic stirrer.
- The potential difference (EMF) was recorded between the calomel and carbon electrodes over a period of time by using a milivoltmeter (mV) till the potential difference attained a nearly constant value.
- The EMF readings are taken at 1 minute time separation up to 30 minutes or till a constant value of EMF is attained.
- The graphs among Time vs. EMF (millivolt) for all the samples are plotted

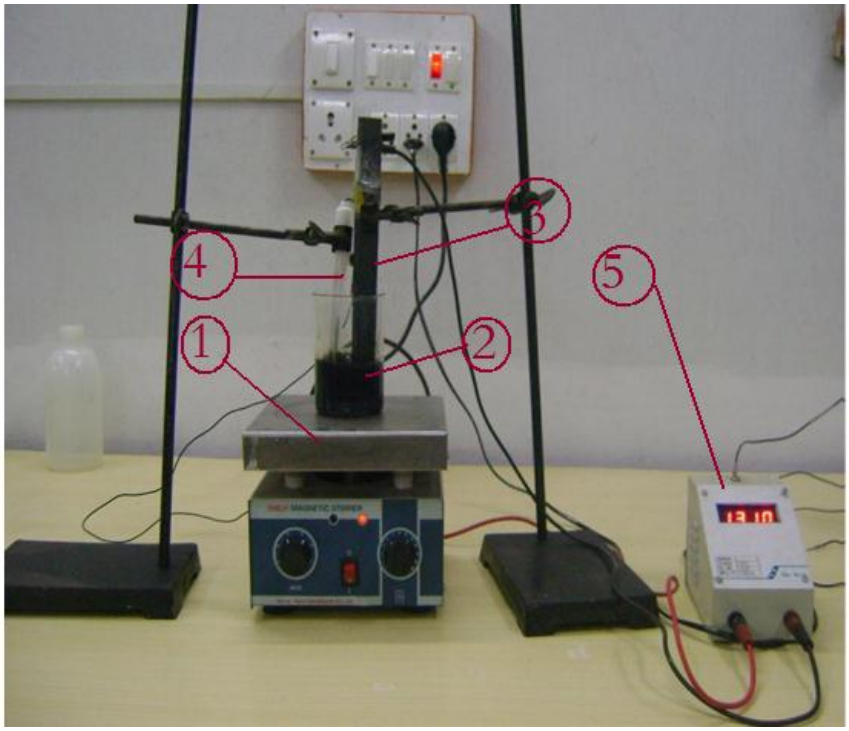


Plate 3.3 Wet oxidation potential apparatus

1. Magnetic Stirrer
2. KMnO4, KOH and Coal Solution
3. Calomel Electrode
4. Carbon Electrode
5. Millivoltmeter

3.3.3 CROSSING POINT TEMPERATURE (Kaymkci et. al., 2000)

This is one of the oldest approach for determining susceptibility towards spontaneous heating of coal. It is the temperature at which the coal temperature coincides with that of the furnace temperature or bath temperature in ^{0}C. In this method, the coal sample is heated in a furnace within a reaction tube at constant rising temperature with oxygen passing through it at a predetermined rate till the coal temperature crosses the furnace temperature.

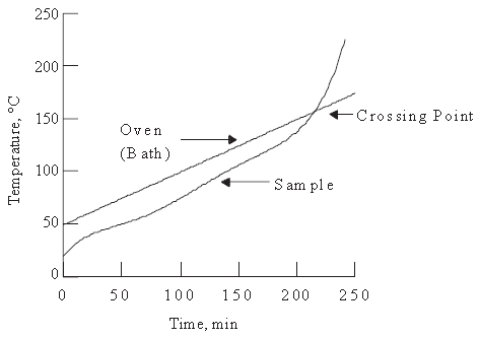


Fig 3.6 Time – Temperature curve for CPT (Didari et.al., 2000)

The experimental setup for the crossing point temperature (CPT) of coal consists of following:

Vertical tubular furnace having heating capacity of 3kw. The furnace is allowed for with a temperature. The length of glass reaction tube is 150mm and the internal diameter of 26mm. The reaction tube has helical glass tube of 6mm internal diameter around it which is connected to the bottom in the inner part of the reaction tube for air inlet and a small out-let tube at the top acts as air/gas outlet. Flow meter and pressure flow control valves. For the removal of moisture from air, a drying tower containing granular calcium chlorides.

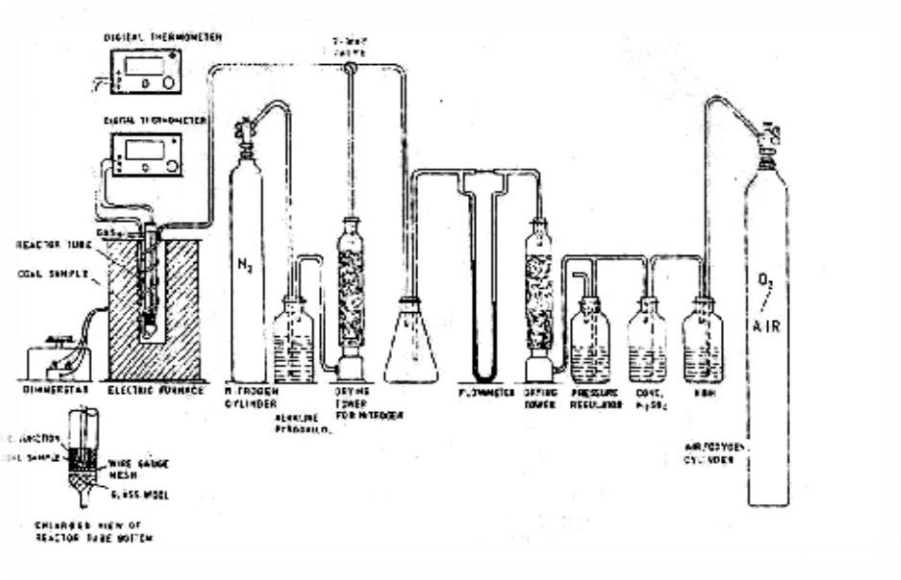


Fig 3.7 Schematic diagram of crossing point temperature (Tripathy et.al., 2001)

Experimental Procedure :

- For this experiment 20gms of coal sample of size -212 micron was placed in the reaction tube where glass wool is followed at the bottom most part of glycerine bath.
- The tube is then lightly tapped with the help of cork, a fixed number of times to achieve uniform packing density of the samples.
- Then the reaction tube is placed in the tubular furnace and a thermocouple is inserted at the centre of the sample for CPT experiment.
- after switching on the furnace air is allowed to pass through the sample simultaneously, with an average heating rate of 1 ^{0}C per minute and air flow is at 80ml/ min. The temperature of the furnace and the coal sample are recorded at every 5 minute interval till the temperature of coal intersected over and gone beyond the furnace temperature.

3.3.4 OLPINSKI INDEX METHOD (Karmakar et.al., 1989)

Reactivity of coal is different temperature. This method determines the reactivity of coal at a temperature of 230 ^{0}C. The reactivity at this temperature does not directly qualify the demeanor of coal at a temperature lying below 70 ^{0}C which is critical for the process of spontaneous combustion. But test has shown that coals having high reactivity index at 230^{0}c energetically react at lower temperature also. Hence susceptibility of coal towards spontaneous heating can be expressed in terms of reactivity of coal in contact with air at a temperature of 230 ^{0}c. In this method liquid is heated in an electric oven to boil gently to produce temperature of 230 ^{0}c quinoline vapour. Small pallets are made of coal powder and heated indirectly by this vapour in an atmosphere of oxygen which passes over the coal pallet at a thermocouple inserted into the coal pallet the temperature of the pallet is noted at a regular interval of time slot the temperature vs time graph. A chart recorder work directly gives the temperature VS time graph. The rate of rise temperature at the moment of equalization of pallet and surrounding temperature i.e. when the whole quantity of heat of reaction is consumed by the pallet for rising its temperature. It is graphically determined by drawing agent to the curve at the point corresponding to the quinoline vapour temperature. The rate of rise of temperature determined in this way is expressed in ^{0}c / min which is an indication of the spontaneous combustibility of coal. This index called as Olpinski Index, is denoted by SZa. Olpinski method makes correction for ash content of coal and express spontaneous combustion index as:

$$SZb = SZa \left(\frac{100}{100 - Aa} \right)$$

Where,

SZa: Olpinski Index

SZb: Spontaneous combustion Index free of ash

Aa: Ash content of coal expressed in percentage(%)

Suscepyibility of coal to spontaneous combustion increases with increase of SZb Index.

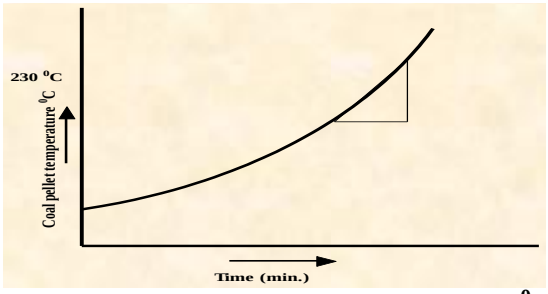


Fig 3.8 Olpinski index curve at 230 ^{0}c

Experimental procedure:

- The 1 gram of coal pallet of -72 mesh (BSS) is taken as the sample for the experiment. The vessel is filled up to the neck with quinoline and placed over the electric oven in a fume chamber.
- The oven temperature is controlled to boil the quinoline (at 230^0c). Oxygen flow is maintained 80 cc per minute and it is controlled by the help of rotametre.
- The thermocouple is inserted in the coal pallet to touch its bottom end. The thermocouple along with pallet is carefully introduced in the combustion tube.
- The reading is taking in the interval of 15 seconds for the better results.
- The temperature of the pallet rises and ultimately it smoulders or even burns out. As soon as smoke is visible at the gas outlet, the thermocouple is taken out.
- Thus for each and every pallet, time vs temperature graph is obtained. Once the quinoline attains its boiling temperature, each pallet takes about 20 to 30 minutes depending on the type of coal.

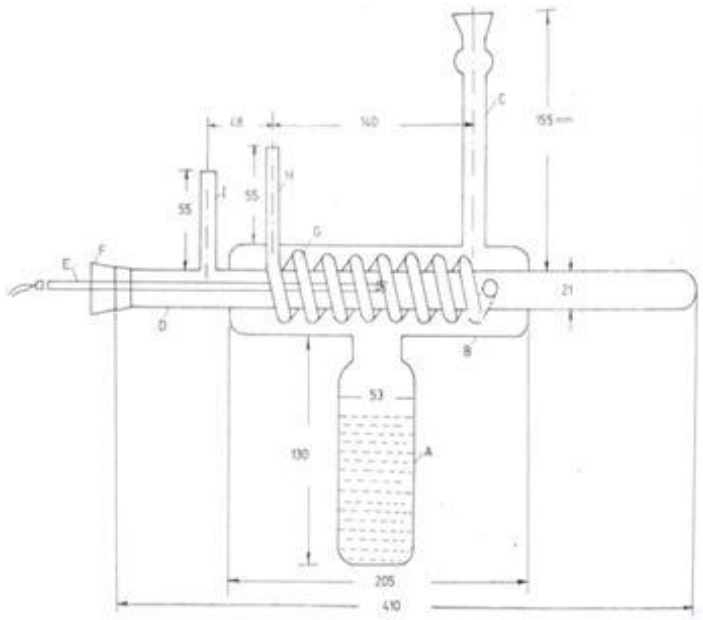


Fig 3.9 Schematic diagram of olpinski index apparatus (karmakar et.al., 1989)

The apparatus consists of a cylindrical glass vessel(A) of volume about 280 cc which is connected to another cylinder (B) perpendicular to it. One funnel (C) is attached to B which also acts as a condenser. A reaction tube (D) passes axially through B which can be heated by quinoline vapour. One end of a thermocouple(E) can be introduced to reach upto the central line of quinoline vessel. A glass tube (G) goes spirally over the reaction tube to release at its

closed end. The combustion product goes out through the outlet (I). The apparatus is mounted over the electric oven to the heat quinoline vessel. The whole set-up placed in a fume chamber with an exhaust fan to clear the quinoline vapour out of the laboratory.

Table 3.2 Classes of coal susceptibility to spontaneous heating (Olpiński et.al., 1952)

Sz_a (^{0}C/min)	Risk Rating
>180	Very Highly Susceptible
80-180	Highly susceptible
40-80	Moderately susceptible
0-40	Poorly susceptible
<80	Poor liability to S.H. risk
>120	High liability to S.H. risk
39-193 ^{0}C/min For Indian coals	

Plate 3.4 olpinski index apparatus

3.3.5 OXYGEN ABSORPTION METHOD / Russian U- Index Method (Karmakar et.al., 1989)

This method is based on absorption of oxygen by coal in static conditions prevalent in CPT or Olpinski Index. In this method a known quantity of crushed coal of particular size range is kept in aglass vessel for a day. Coal powder absorbs oxygen to foem a coal – oxygen-complex and gives out CO_2. If the coal contains methane, it is desorbed in the vessel is analysed for CO_2, CH_4 & O_2 concentration to calculate the oxygen absorbed by coal which is

expressed in ml of oxygen absorbed per hour per gm of coal(Ml/Hr/Gm). This is known as oxygen absorption by the coal is more *i.e* if the U-index is high, the coal is more prone to spontaneous heating. U –index is given by the formula:

$$U = \frac{-V\ (B-P)}{W*T*760} Ln\ \frac{(1-C_0)*C_a}{C_0*(1-C_a)}$$

Where v: volume of air enclosed in the vessel (ml)

B: Barometric Preassure (mm Hg)

P: Saturation Vapour preassure at room tempreture (mm Hg)

W: Weight of coal sample (g)

T: Time of absorption (hr)

C_a: Concentration (in decimal) of oxygen in the air inside the glass after time T and is given by

$$C_a = \frac{O_2}{1-CO_2-CH_4}$$

Where, O_2, CO_2, and CH_4 represent the concentration of the respective gases.

760: Barometric preassure at mean sea level (mm Hg).

EXPERIMENTAL SET-UP:

The apparatus consists of cylindrical glass vessel of about 600 ml volume with wide mouth. The lower part of the vessel is designed in a particular fashion. At the end of the cylindrical portion it becomes narrow and then forms a small bulb of volume about 30 ml and again becomes narrow. The function of the small bulb is to act as a water reservoir thus preventing the coal powder from getting wet.

The mouth vessel can be closed bya glass stopper with an in-built arrangement for drawing gas sample from it to a sample collector for analysis. The bottom end of the vessel ids dipped into a water through. The water is made slightly acidulated by dissolving sodium solphite ($Na_2\ So_3$) in it.

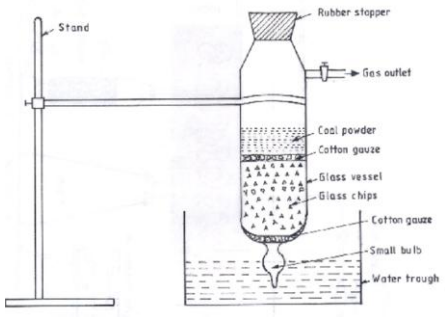


Fig 3.10 Schematic diagram of Oxygen absorption apparatus (karmakar et.al., 1989)

Solubility of carbon- dioxide is negligible iv such acidulated water. The vessel is clamped onto a stand to hold it in vertical position.

Experimental procedure:

A piece of cotton gauge is insertwd into the vessel to cover the narrow opening above the small bulb. 200 g of glass chips is taken in the vessel (glass rod about 5 mm diameter is broken in to pieces of about 1 cm long to form glass chips). The glass chips bed is covered by another piece of cotton gauge. 40 g of coal sample is size between 1 mm and 3.175 mm (1/8") is poured into the vessel to form a thin layer over the glass –chip bed. The mouth of vessel is closed by stopper applying some vesline to make it air tight. The bottom and of the vessel is slightly dipped into the sodium-sulphite solution in the through and secured vertical position by clamping it onto a stand. After a period of 24 hours, the sample of gas above the coal bed is drawn in gas sample collector by water displacement. Instead of pure water, sodium sulphite solution is used. The gas sample is then analysed by Haldane Apparatus to determine the concentration of O_2, CO_2, and CH_4 . the accuracy of the apparatus is 0.01 ml.

3.3.6 Differential thermal analysis (Nimaje et.al., 2010)

DTA is thermoanalytic technique, in DTA, the material under study and inert temperature difference between sample and reference. This differential temperature is plotted against time, or against temperature (DTA curve or thermogram). In differential thermal analysis, the difference in temperature between a substance and a thermally inert reference material against temperature is recorded as the two specimens are subjected to identical temperature exchanges in a block which is heated at a linear heating rate.

The record is the differential thermal or DTA curve; the temperature difference should be plotted on the ordinate with endothermic reactions downwards and temperature or time on the abscissa increasing from left to right.

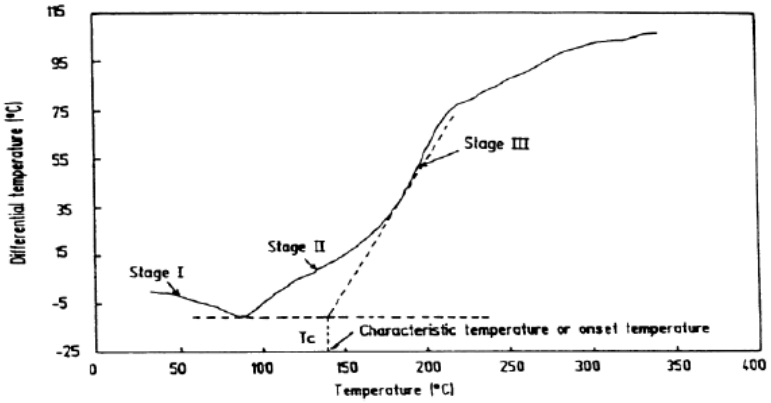


Fig 3.11 Different stages of DTA Nimaje et.al., 2010)

Changes in the sample can be observed relative to the inert reference by either exothermic or endothermic reaction. Thus DTA curve furnishes data on the transformations that have took place. The area under a DTA peak is enthalpy change is not involved by heart capacity of the sample. The DTA apparatus consists of a sample and reference holder, a furnace, a temperature programmer to maintain constant temperature rate and an output to monitor the test. The sample holder has two thermocouples one for coal sample and another for reference material. The sample is contained in a small crucible By the temperature programmer the temperature is made to rise at a constant rate and the temperature difference of the sample and reference is plotted against time. The plot consists of three parts – stage-I, II, III. The temperature falls down due to release of moisture during stage-I because mostly endothermic reactions take place. Stage II consists of two parts such as stage-II A and stage-II B. At the starting of stage-II A the heating tendency starts accompanied by a small amount of endothermic reactions. The point where stage-II A begins is called as the inflexion point as the thermo gram suddenly rises. In stage-II B exothermic reactions start to prevail. The beginning of stage-III is called as Transition temperature. This temperature is very significant as it is directly related to spontaneous heating tendency of coal. The lower the transition temperature the higher is the susceptibility of coal and vice versa. From stage-III onwards complete exothermic reactions placed and the temperature preserves to rise steeply.

Plate 3.5 Experimental Set up of Differential Thermal Analysis Apparatus

Experimental Procedure:

- The furnace is lifted by controlling the switch and the crucible for sample and reference were put in position on the post so that thermocouple tips of both the sample holders were just below them
- About 10 mg of -212 micron (BSS) coal sample is taken in the crucible provided with the apparatus.
- Oxidising atmosphere was preserved by keeping the coal sample exhibited to air.

- The reference material taken is alpha alumina (Al_20_3) and atmospheric air is taken as medium.
- The DTA machine is switched on and the software is set according to required conditions viz., heating rate is maintained at 5 ^{0}C/minute and the final temperature is taken as 450 ^{0}C.
- After the heating programme was terminated, thermo gram is obtained for coal sample.
- The plots found are then analysed for the slopes of various stages and transition temperature.

3.3.7 DIFFERENTIAL SCANNING CALORIMETRY (Mohalik et.al., 2009)

Differential scanning calorimetric is subjected to a controlled temperature program in this technique the difference in energy in puts into a substance and a reference material is measured as a function of temperature while the substance and reference materials. In this technique the ordinate value of an output curve at any given temperature is directly proportional to the differential heat flow between a sample and reference material and in which the area under the measured curve is directly proportional to the total differential calorific input. Coal samples could be studied under experimental conditions that for assumption of spontaneous combustion process of materials by this technique. The complete experimental set up comprises the different parts of differential scanning calorimeter (DSC), sample holder, crucible sealing press, purge gas supply arrangement, a computer with software and a graphic plotter for the graph. In DSC therefore, endothermic responses are usually represented as being negative, i.e. below the baseline, corresponding to an increased transfer of heat to the samples compared to the reference. Purge gas is supplied from a cylinder equipped with suitable regulators. All the operations of the DSC calorimeter are controlled from the personal computer through software. The software performs all the controls, calibration, data display, standard calculations, curve comparison and calculations etc.

Experimental procedure:

DSC apparatus was calibrated as for the sample via the melting points of indium, zinc, lead metals under the same conditions. At about 10mg coal sample of -212 micron was taken in the perforated aluminum sample pans and weighed accurately using a single pan electronic balance . The sample was subjected to a computer controlled temperature programme, which is a software that the heat flow of time and temperature were recorded simultaneously, during the experiments. By the help of crimper press sample was placed in the sample pan, covered with a lid and sealed with pressure. After that crimped container with the sample was put on

the sample furnace while the reference furnace was kept empty. For good heat transfer, the coal was placed in a monolayer at the base of the pan to ensure that the entire sample made good thermal contact with the bottom part of the pan with the sensors of the equipment. If the pinholes were much smaller then the oxygen diffused into the pans were too slowly and by this process determined the rate of oxidation, rather than diffusion into the pore structure of the coal itself. A number of experiments were contained in the temperature range from ambient (30 ^{0}C) to 600 ^{0}C with changing atmosphere. To check the duplicability of the results, the experiments were repeated under identical conditions.

Table 3.3 DSC Experimental Parameters Studies on S H (Mohalik et.al., 2009)

Si.no	Name of the Author	Year	Parametres							
			Paricle Size(mesh)	Heating rate (^{0}c/min)	atmosphere	Samole amount (mg)	Flow rate (ml/min)	No of Sample Studied	Reference material	Temp ^{0}c
1.	Mahajan, Tomita & Walker(1976)	1977	-	10	Heleium	20,12	1	12	Alumina	100 to 580
2.	Ismail & walker(1989)	1989	-100	10	N_2	-	45	16	-	Ambient to 600
3.	Kok(1997)	1997	-60	5,10,15, 20,25	Air	-	50	1	-	20 to 600
4.	Garcia, Halla&Fadoor (1999)	1999	- 100	10	O_2	10	20	3	-	Ambient to 600
5.	Ozbas,kok& Heiyillmaz	2003	-	` 10	Air	10	50	4	-	20 to 600
6.	Panigrahi& Sahu	2004	-72	30	O_2	10	20	31	Alumina	Ambient to 500
7.	kok	2005	-60	10	Air	10	50	17	-	20 to 600
8.	Elbeyli& Piskin	2006	-60	10	Air N_2	10	100	1	-	Ambient to 1000
9.	Krzesinska et,al	2008	-	10	N_2	7	50	3	-	Ambient to 520

CHAPTER – 4

RESULTS AND ANALYSIS

ABSTRACTS OF EXPERIMENTS

1. Proximate analysis (IS 1350 part-1, 1984)

A. Determination of moisture :

- Amount of coal : 1 g coal
- Size of coal : - 212 micron (-72 mesh)
- Heating time : 1.5 hours at 110^0 C

B. Determination of volatile matter :

- Amount of coal : 1 g of coal
- Size of coal : - 212 micron (-72 mesh)
- Heating time : 7 minutes at 900^0 C

C. Determination of ash :

- Amount of coal : 1 g of coal sample
- Size of coal : - 212 micron (-72 mesh)
- Heating time : 30 minutes at 450^0 C and 60 minutes at 850^0 C

2. Wet oxidation potential study (Tarafdar et.al., 1989)

- Amount of coal : 0.5 g of coal sample
- Size of coal : -212 micron(-72 mesh)
- System : Coal + 1 N KMnO4 + 0.1 N KOH)= 100 ML

3. Flammability temperature (Nimaje et.al., 2010)

- Amount of coal : 200 mg of coal sample
- Size of coal : -200 mesh (BSS)
- System : Coal + Air
- Volume of air : 500 ml
- Pressure of air : 8 cm of Hg

4. DTA (Nimaje et.al., 2010)

- Amount of coal : 10 mg of coal sample
- Size of coal : - 212 micron (BSS)
- Heating rate : 5^0 C/min

- Hold time: 450^0 C
- System : coal + air

5. Bomb Calorimeter (IS 1350-1959)

- Amount of coal : 1gm of coal sample
- Size of coal : - 212 micron (-72 mesh)
- pressure :- 300 - 400 PSI

6. Olpinski Index (Karmakar et.al ,1989)

- Amount of coal : 0.3 – 0.4gm of coal sample
- Size of coal : -200 B.S mesh
- Heating rate : 235^0 C/min
- Air flow rate : 200-300 ml/min

Table 4.1 List of coal samples

Sl. No.	Sample	Name of the organization
1	MCL 1	MCL
2	MCL 2	
3	MCL 3	
4	MCL 4	
5	MCL 5	
6	MCL 6	
7	,MCL 7	
8	MCL 8	
9	NCL 1	NCL
10	NCL 2	
11	IISCO 1	IISCO
12	IISCO 2	
13	IISCO 3	
14	IISCO 4	
15	BCCL 1	BCCL
16	TISCO 1	TISCO

Table 4.2 Results of proximate analysis parameters

Sample	Moisture	Ash Content		Volatile matter			Fixed carbon		
	ar	ar	db	ar	db	daf	ar	db	daf
MCL-1	7.1	18.5	19.91	33.44	50.24	44.94	40.96	48.06	66.56
MCL-2	14.37	13.9	16.23	31.82	46.67	44.36	39.91	54.28	6818
MCL-3	2.71	13.53	13.90	33.24	49.79	39.68	50.52	53.23	66.76
MCL-4	15.37	6.5	7.68	22.88	29.66	29.28	55.25	70.62	77.12
MCL-5	12.13	14.8	16.84	34.12	51.79	46.69	38.95	51.08	65.58
MCL-6	13.67	13.89	16.08	31.19	45.32	43.05	41.25	54.92	68.81
MCL-7	19.37	15.5	19.22	32.45	48.03	49.82	32.68	52.05	67.55
MCL-8	15.36	8.9	10.51	31.10	45.13	41.06	44.64	60.00	68.9
NCL-1	15	15.39	18.10	25.72	34.62	36.94	43.89	58.89	74.28
NCL-2	11.80	11.14	12.63	26.49	36.03 .	34.37	50.57	62.37	73.51
IISCO-1	1.17	5.42	5.48	39.89	66.36	42.70	53.52	54.69	60.11
IISCO-2	1.5	6.63	6.73	41.27	70.27	44.92	50.60	52.10	58.73
IISCO-3	1.6	1.63	1.65	42.87	75.03	44.30	53.90	55.50	57.13
IISCO-4	1.65	12.26	12.45	40.94	69.31	47.55	45.15	46.80	59.06
BCCL-1	1.53	4.82	4.89	38.15	61.68	34.00	55.50	57.03	61.85
TISCO-1	3.8	5.8	6.02	43.79	77.90	48.44	46.61	50.41	56.21

Where,

Ar- as received

Db- dry basis (100/100-M)

Daf – dry ash free basis (100/100-(M+A))

Dmmf- Dry mineral matter free basis-(100/100-(M+VM))

Table 4.3 Results of calorific value

SAMPLE	GCV (kCal/kg)	UHV (kCal/kg)	GRADE
MCL-1	4319	5367.2	D
MCL-2	3445	4998.7	E
MCL-3	6564	6658.8	A
MCL-4	5750	5881.9	C
MCL-5	4406	5183.6	D
MCL-6	4635	5109.1	D
MCL-7	2547	4087.9	F
MCL-8	5960	5552.1	B
NCL-1	3687	4706.1	E
NCL -2	6728	7114.3	A
IISCO-1	6902	7990.6	A
IISCO-2	6814	7778.1	A
IISCO-3	7010	8454.3	A
IISCO-4	6313	6980.4	A
BCCL-1	7054	8023.7	A
TISCO-1	7222	7575.2	A

Table 4.4 Results of wet oxidation potential

Sample	EMF (mV)
MCL-1	133
MCL-2	152
MCL-3	155
MCL-4	134
MCL-5	167
MCL-6	139
MCL-7	156
MCL-8	117
NCL-1	182
NCL-2	148
IISCO-1	116
IISCO-2	114
IISCO-3	104

IISCO-4	111
BCCL-1	108
TISCO-1	101

Table 4.5 Results of flammability temperature

Sample	Flammability Temperature (^{0}C)
MCL-1	580
MCL-2	500
MCL-3	560
MCL-4	500
MCL-5	540
MCL-6	540
MCL-7	480
MCL-8	540
NCL-1	490
NCL-2	530
IISCO-1	580
IISCO-2	500
IISCO-3	560
IISCO-4	480
BCCL-1	540
TISCO-1	550

Table 4.6 Results of DTA – TG

Sample	Stage IIA	Stage IIB	Stage II	Transition Temperature (^{0}C)
MCL-1	0.374	0.805	0.530	178.60
MCL-2	0.385	0.037	0.584	178.26
MCL-3	0.387	0.690	0.516	159.35
MCL-4	0.495	0.242	0.534	098.78
MCL-5	0.310	0.084	0.201	125.10
MCL-6	0.325	0.368	0.346	153.08

MCL-7	0.100	0.181	0.142	146.76
MCL-8	0.891	0.089	0.985	152.16
NCL-1	0.182	0.883	0.997	144.47
NCL-2	0.665	0.783	0.717	160.06
IISCO-1	-0.032	0.141	0.123	152.70
IISCO-2	0.035	0.548	0.100	247.57
IISCO-3	0.110	-0.076	0.072	208.48
IISCO-4	0.091	0.751	0.198	221.61
BCCL-1	0.038	0.724	0.148	247.14
TISCO-1	0.050	0.801	0.148	239.44

Table 4.7 Result of olpinski Index

SAMPLE	SZ_a (^{0}C/min)	SZ_b (^{0}C/min)
MCL-1	4.76	0.058
MCL-2	3.5	0.040
MCL-3	4.09	0.047
MCL-4	4.16	0.044
MCL-5	5.56	0.065
MCL-6	5.52	0.064
MCL-7	4.59	0.054
MCL-8	4.41	0.048
NCL-1	3.93	0.046
NCL-2	4.73	0.053
IISCO-1	3.56	0.037
IISCO-2	5.00	0.053
IISCO-3	3.94	0.040
IISCO-4	4.45	0.050
BCCL-1	4.71	0.049
TISCO-1	4.41	0.046

Analysis :

The comparison were done in between coking and non-coking coal of different samples by using the different experiments for the determination of spontaneous heating. The various graph plotted by the Excel is shows the comparison between the intrinsic properties of coal and various susceptibility indices and also the comparison among all the susceptibility indices of coal sample.

CHAPTER – 6

DISCUSSION AND CONCLUSIONS

Discussion:

The susceptibility of coal to spontaneous combustion depends on its intrinsic properties. Therefore, the comparison have been carried out between the different susceptibility indices such as wet oxidation potential, differential thermal analysis and flammability temperature, olpinski index. The susceptibility indices are taken as dependent variables and each constituent obtained from the proximate and ultimate analyses as an independent variable. In general following things are to be noted from the results of all experiments:

- The highest moisture content was found to be in MCL-7 which is 19.37% as the seam is watery in nature.
- Field observation shows that mcl-7 is watery in nature and compared with the experimental results.
- MCL-1 has the highest ash content but MCL-7 has low FC content and hence these are high susceptibility for spontaneous heating.
- MCl-7 has highest moisture content and low FC content showing that it is highly susceptible.
- TISCO-1 with highest VM content and MCL-7 has lowest FC content which showing the highest susceptibility for spontaneous heating.
- IISCO-3 has highest FC and low moisture content showing that it is highly susceptible.
- TISCO-1 has the highest GCV (higher rank coal) which shows that it has low susceptibility towards spontaneous heating.
- MCL-2, MCL-7 and NCL-1 have low GCV value which shows that they are lower rank coals and have higher susceptibility than other coals.
- NCL- 1 has the highest EMF values with 182 mV which shows that it is highly susceptible.

- TISCO-1 has the lowest EMF values with 101 mV which shows that it is lowly susceptible.
- The lowest transition temperature was found to be for MCL-4 showing it has the highest susceptibility for spontaneous heating and highest was for IISCO-2 indicating it to be least susceptible.
- DTA results show that BCCL-2 is the moderately high susceptible sample but the transition temperature is also high.
- TISCO -1 is the least susceptible with a reasonably high transition temperature.
- The slope for stage-IIB was found to be maximum for NCL-1 and lowest for IISCO-3.
- The slope for stage-II was found to be maximum for NCL-1 and lowest for IISCO-3.
- MCL-7 and IISCO-4 has lower flammability temperature, high susceptible towards spontaneous heating.
- MCL-1 and IISCO-1 has high flammability temperature, low susceptible towards spontaneous heating, such as MCL-3 & TISCO-1 are moderately susceptible.

Conclusion :

- Wet oxidation potential difference method is good for predicting the spontaneous heating susceptibility of low and high moisture coal.
- Spontaneity increases with increase of moisture content and is the highest for moderate range (4-7%), after which it gradually declines.
- ΔE for one coal sample can be determined in less than 30min.,whereas, the determination of CPT takes approximately 2-3 hours time.
- Olpinski method gives excellent repeatability of the experimental results for the same coal sample, but in case of CPT.
- ΔE method is more handy, quick and easy to perform compared to CPT.
- Determination of Olpinski index is much quicker and it gives a better range for weathered coal compared to CPT, whereas it takes only 20-30minutes for each sample.
- The DTA parameters show a very good parameters which indicates that they predict the spontaneous heating susceptibility of coal better than Wet oxidation potential, Flammability temperature and Calorific value of coal.

- The Transition temperature of DTA is used to measure the susceptibility of coal for spontaneous heating, but Stage-IIB shows better values and hence this may be considered as a better parameter for the measure of susceptibility of coal.
- Flammability temperature could not be taken as a good parameter for evaluating spontaneous heating susceptibility of coal as it showed poor correlation with all the parameters.
- It is observed from the above observations that Differential Thermal Analysis (DTA) predicts the spontaneous heating susceptibility more accurately than Wet Oxidation potential and Flammability Temperature method.
- The few more remaining experiments are only clear the methods significant/insignificant to evaluate the spontaneous heating susceptibility of coal.

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APPENDIX

APPENDIX 1 – WET OXIDATION CURVE

APPENDIX 2 – OLPINSKI INDEX CURVE

APPENDIX 3 – DTA-TG CURVE

APPENDIX 1

WET OXIDATION CURVES

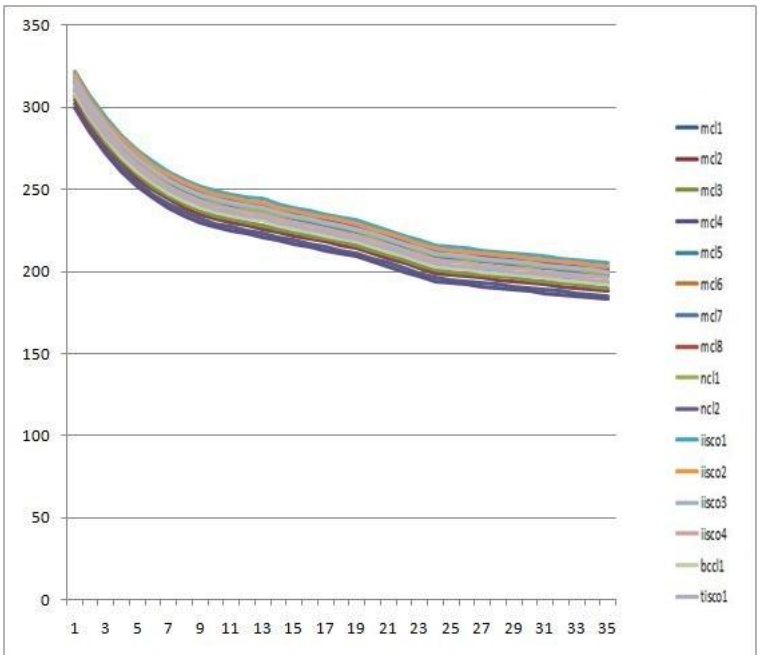

Fig A1 Wet oxidation potential curve for the samples

APPENDIX 2

OLPINSKI INDEX CURVE

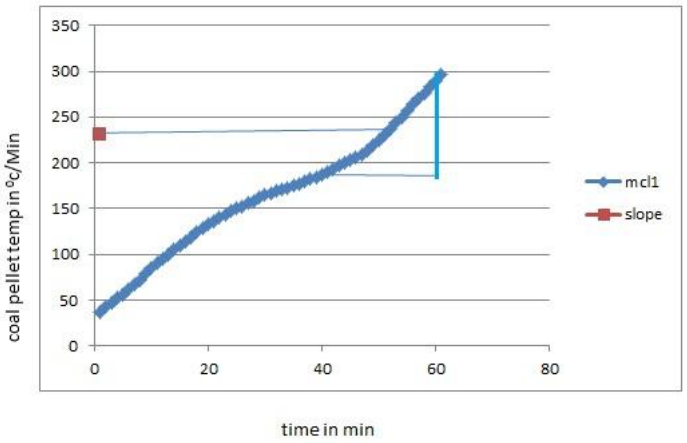

Fig A2 for olpinski index curve of MCL-1

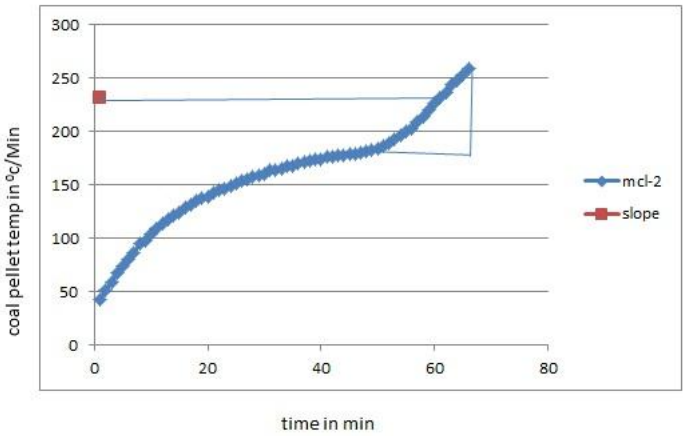

Fig A3 for olpinski index curve of MCL-2

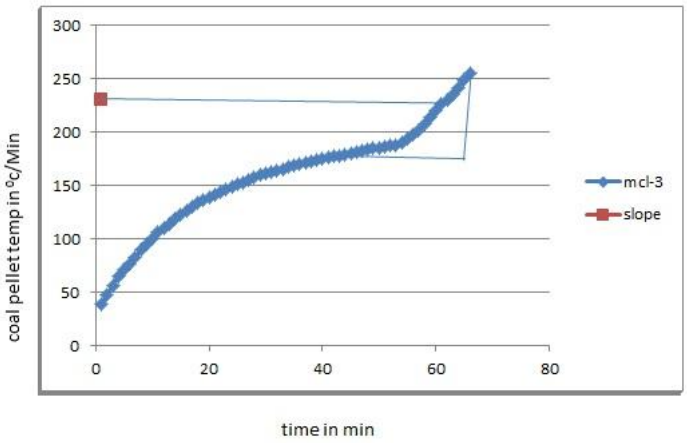

Fig A4 for olpinski index curve of MCL-3

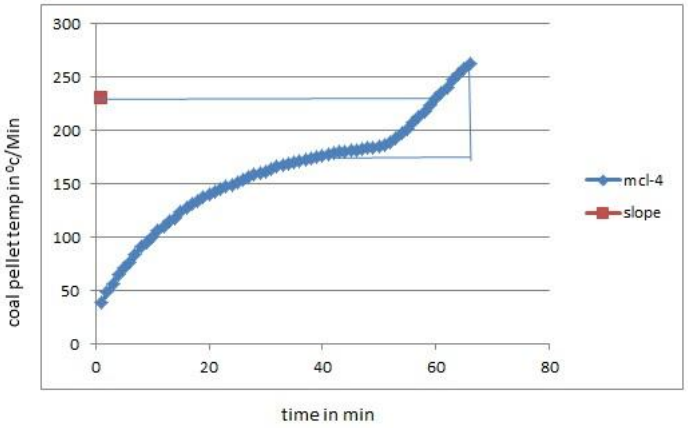

Fig A5 for olpinski index curve of MCL-4

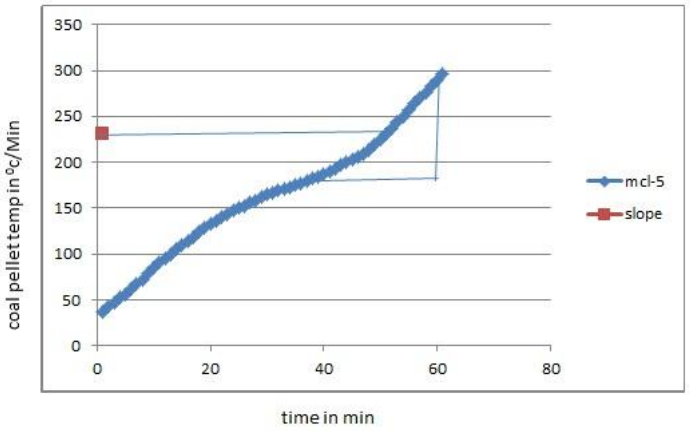

Fig A6 for olpinski index curve of MCL-5

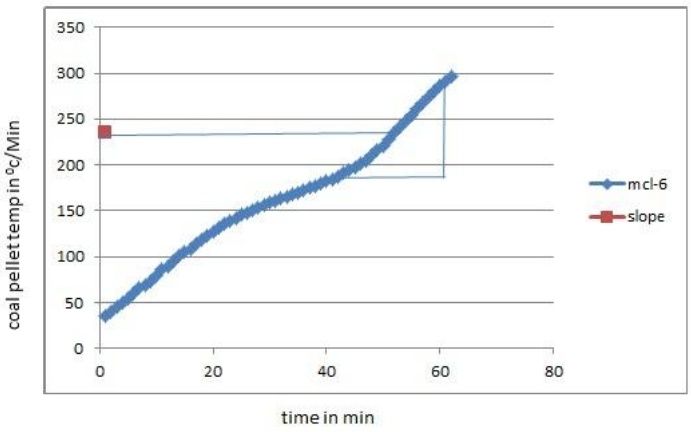

Fig A7 for olpinski index curve of MCL-6

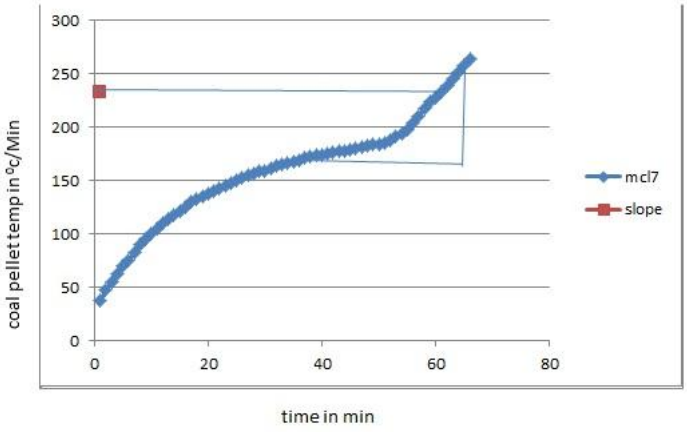

Fig A8 for olpinski index curve of MCL-7

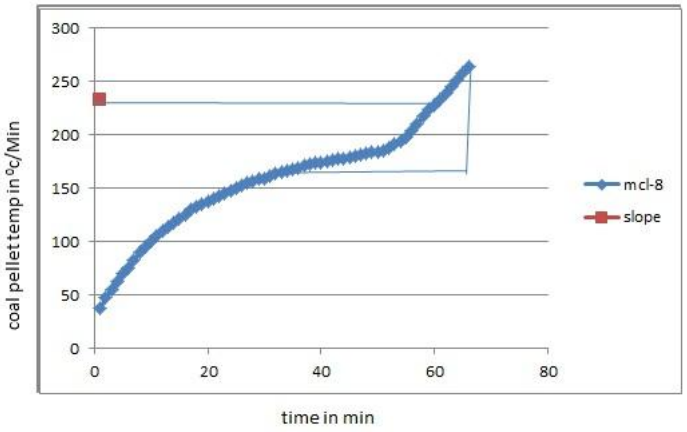

Fig A9 for olpinski index curve of MCL-8

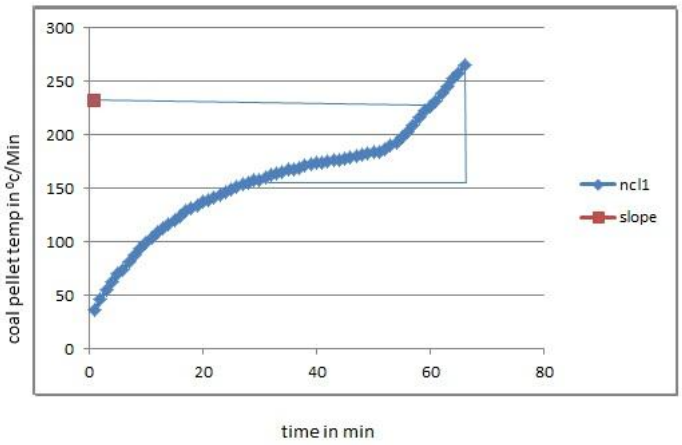

Fig A10 for olpinski index curve of NCL-1

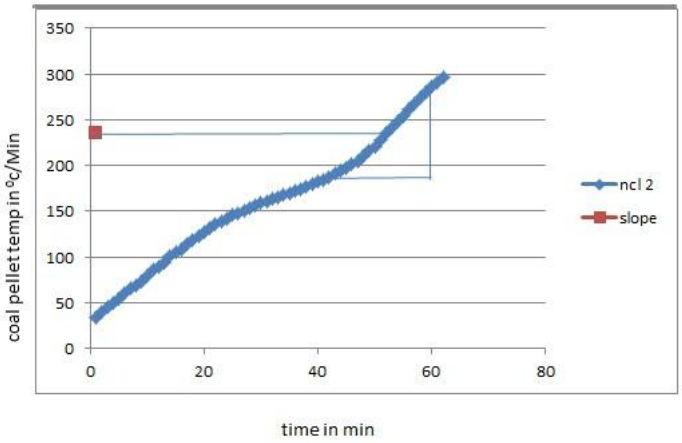

Fig A11 for olpinski index curve of NCL-2

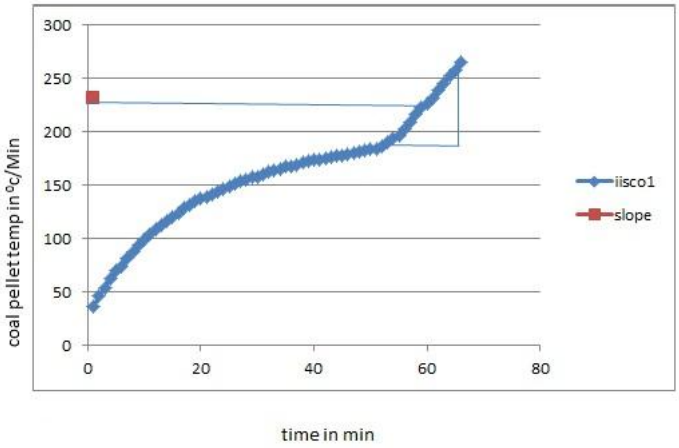

Fig A12 for olpinski index curve of IISCO-1

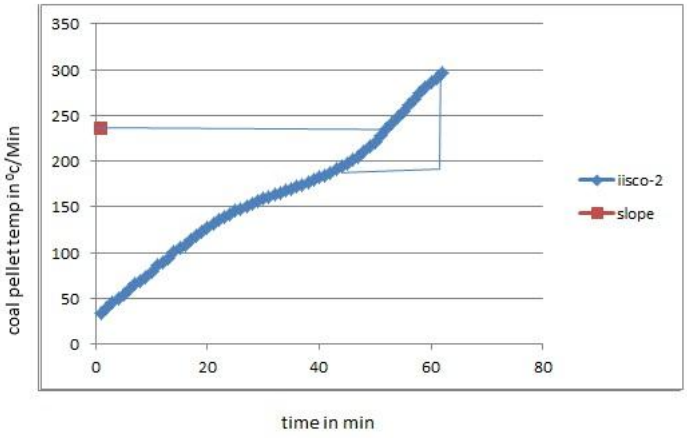

Fig A13 for olpinski index curve of IISCO-2

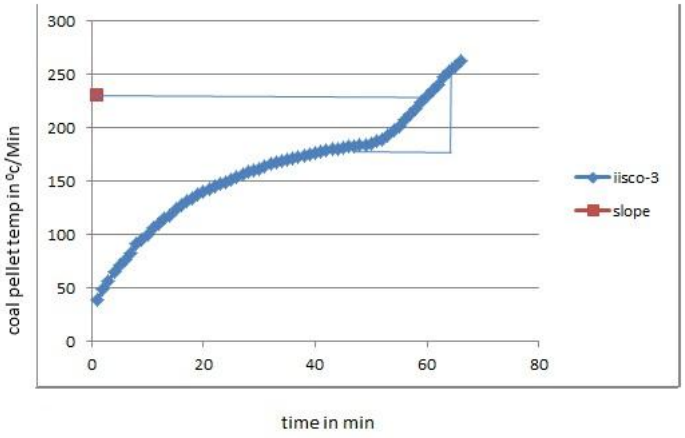

Fig A14 for olpinski index curve of IISCO-3

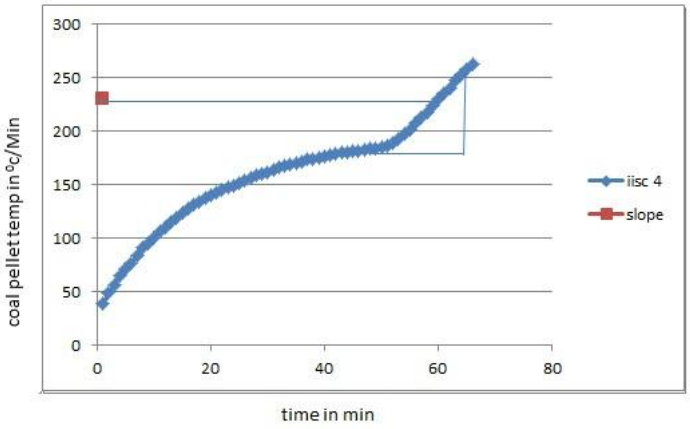

Fig A15 for olpinski index curve of IISCO-4

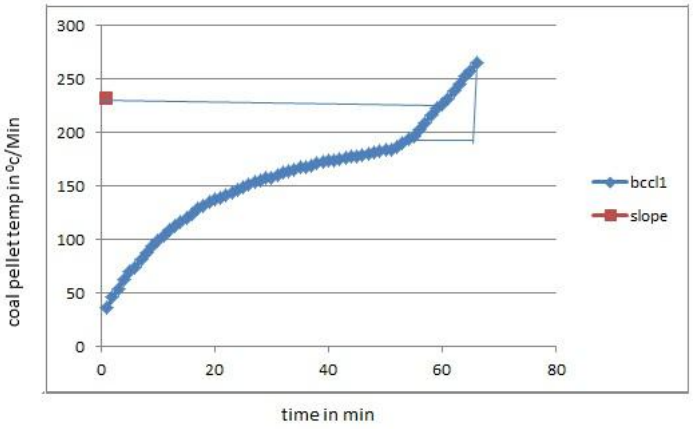

Fig A16 for olpinski index curve of BCCL-1

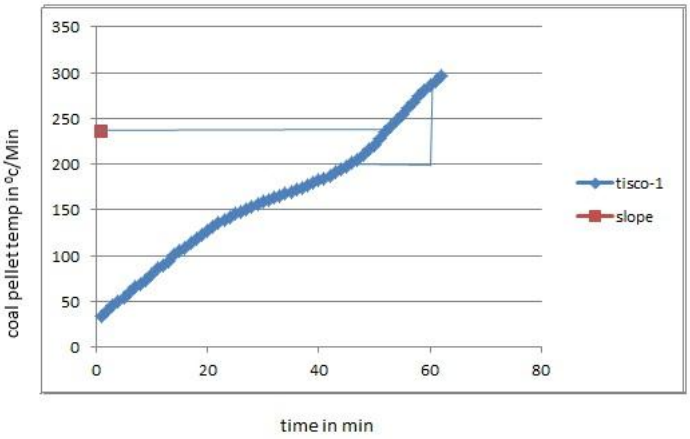

Fig A17 for olpinski index curve of TISCO-1

APPENDIX -3

DTA-TG CURVE

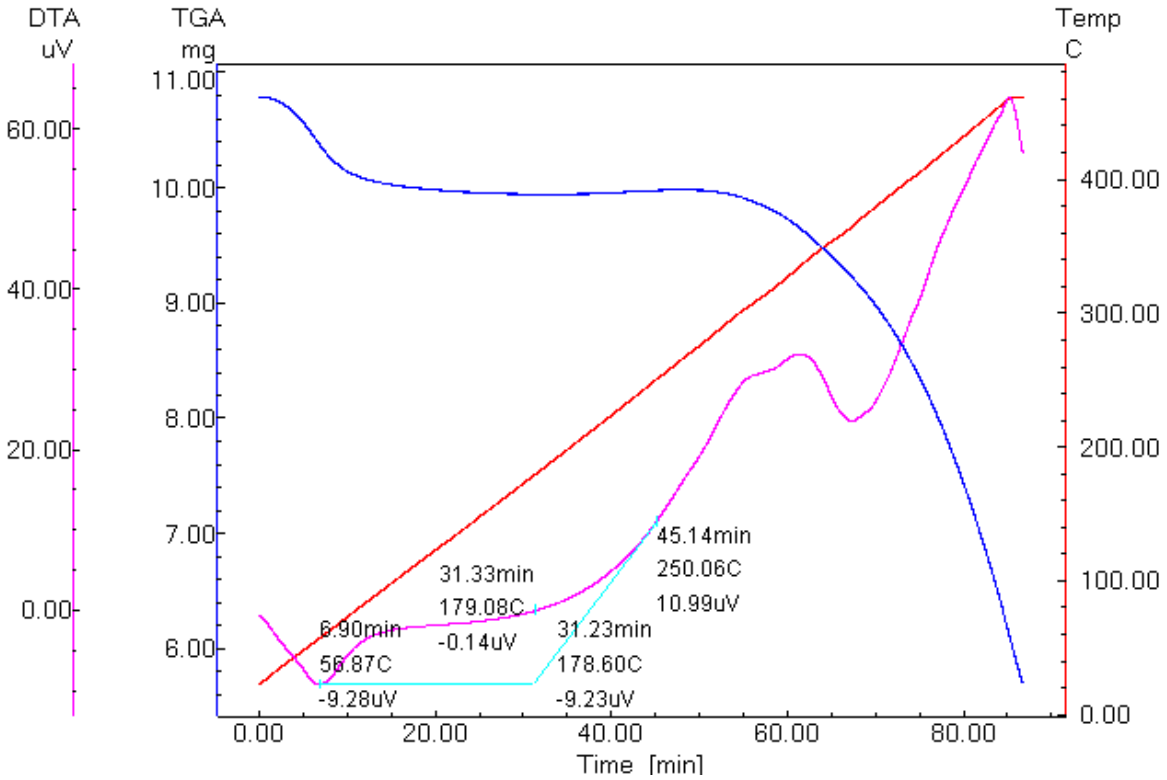

Fig A18 DTA – TG curve of MCL-1

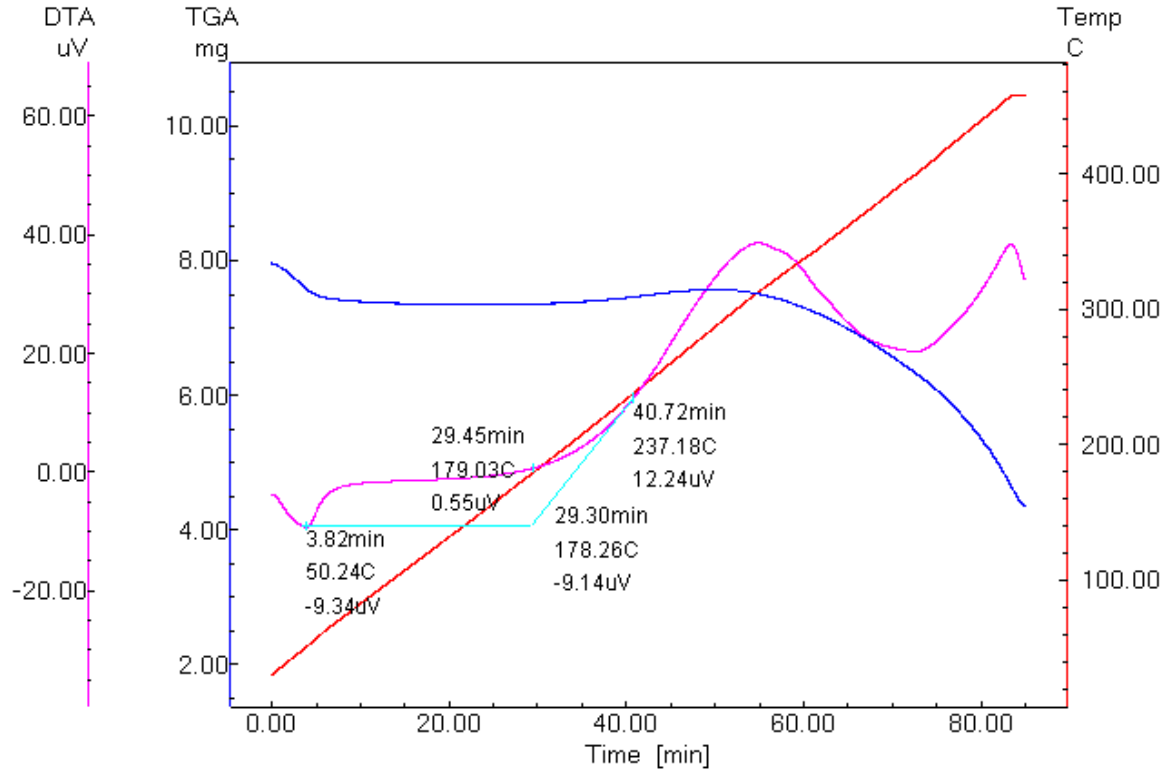

Fig A19 DTA – TG curve of MCL-2

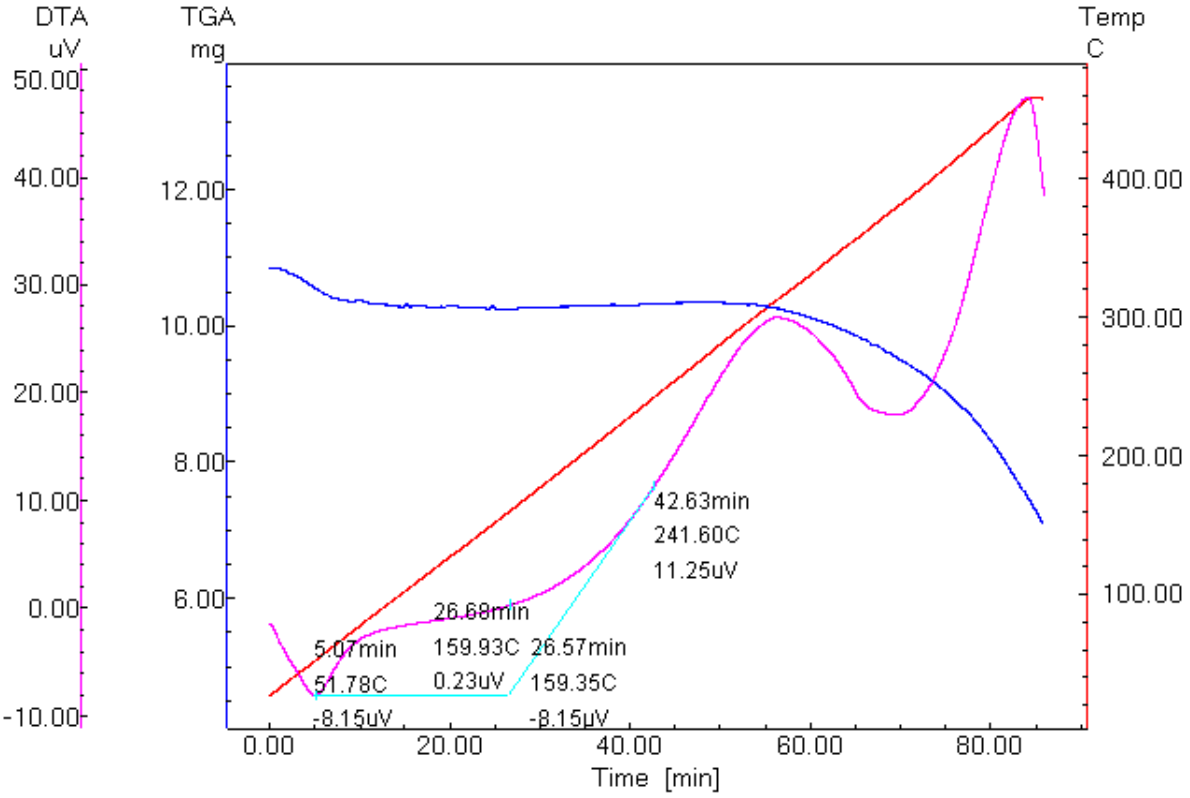

Fig A20 DTA – TG curve of MCL-3

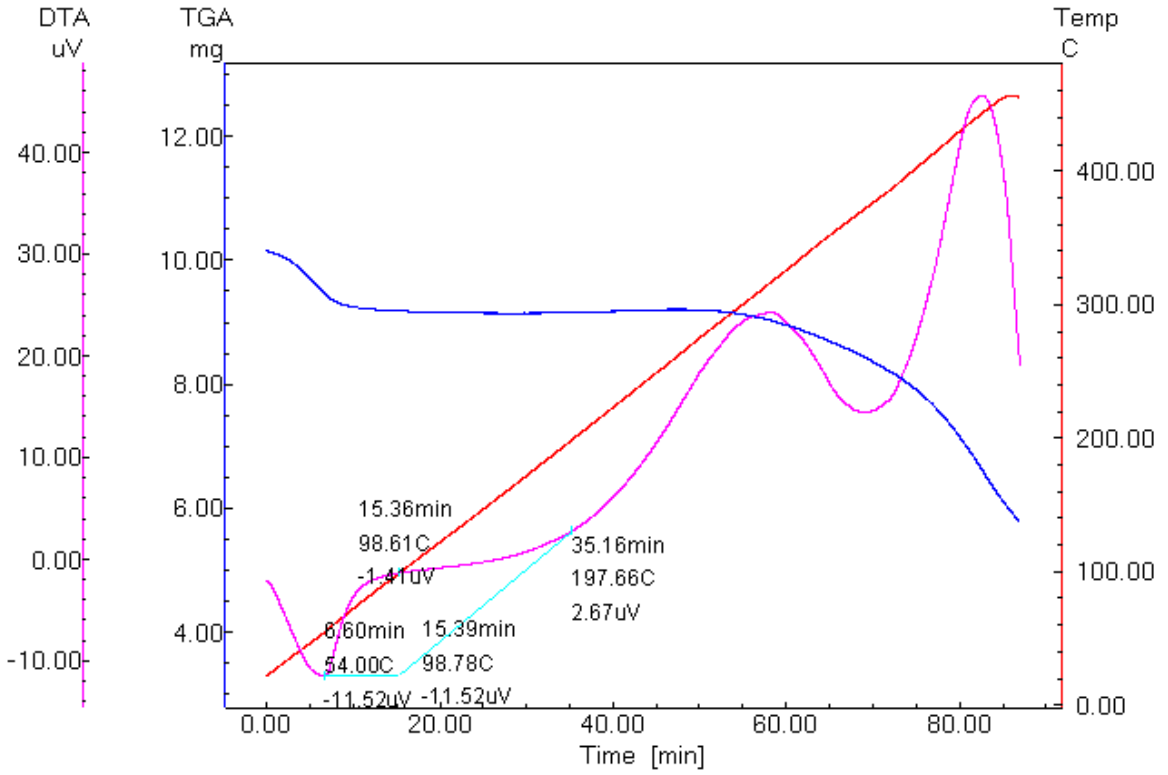

Fig A21 DTA – TG curve of MCL-4

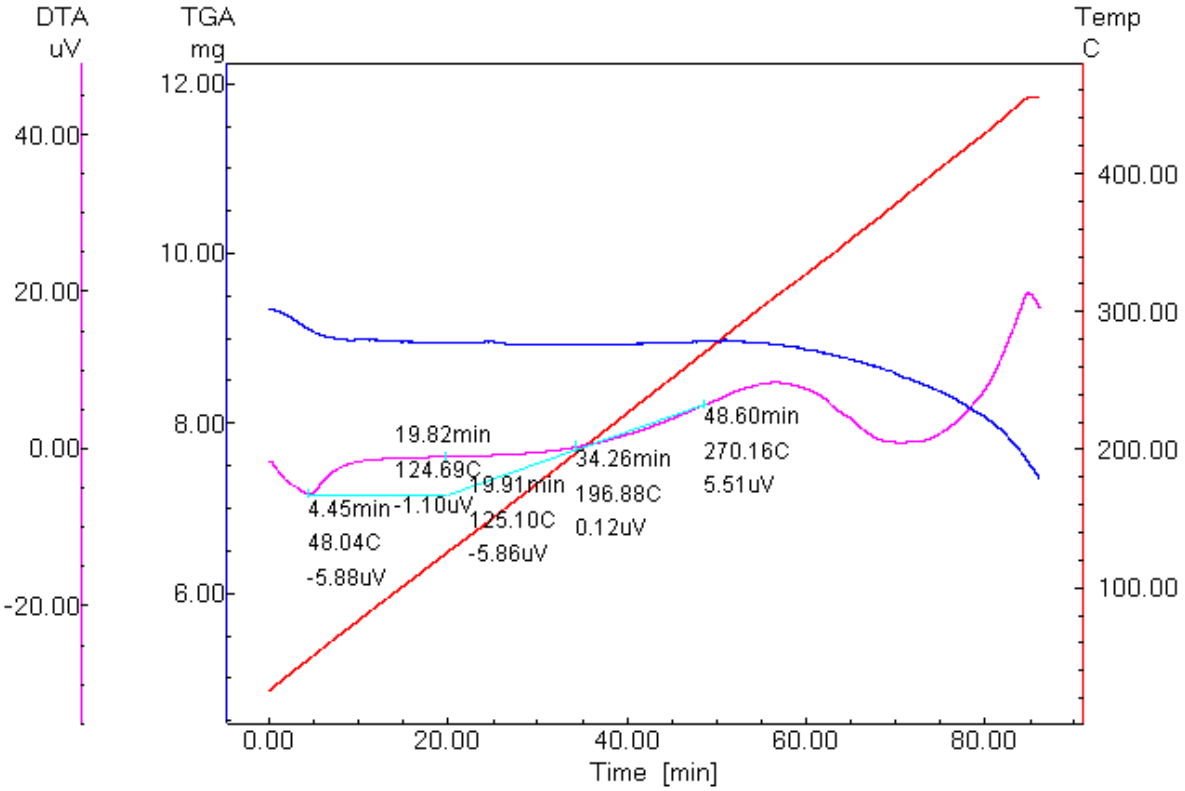

Fig A22 DTA – TG curve of MCL-5

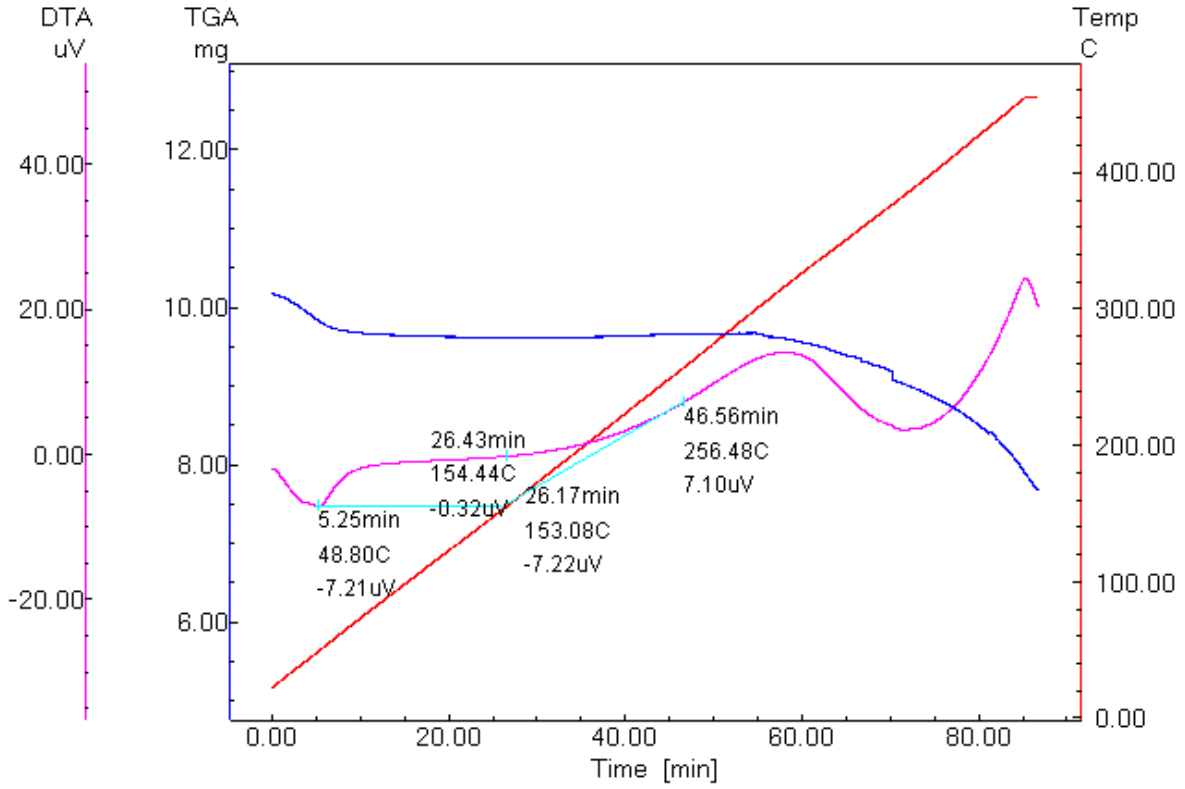

Fig A23 DTA – TG curve of MCL-6

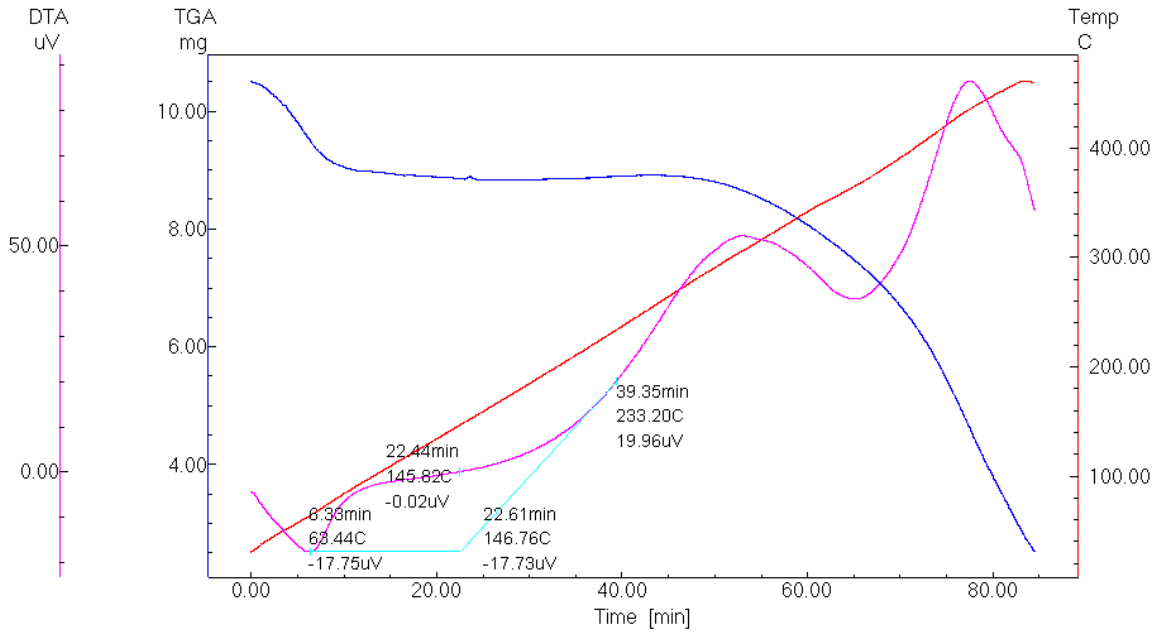

Fig A24 DTA – TG curve of MCL-7

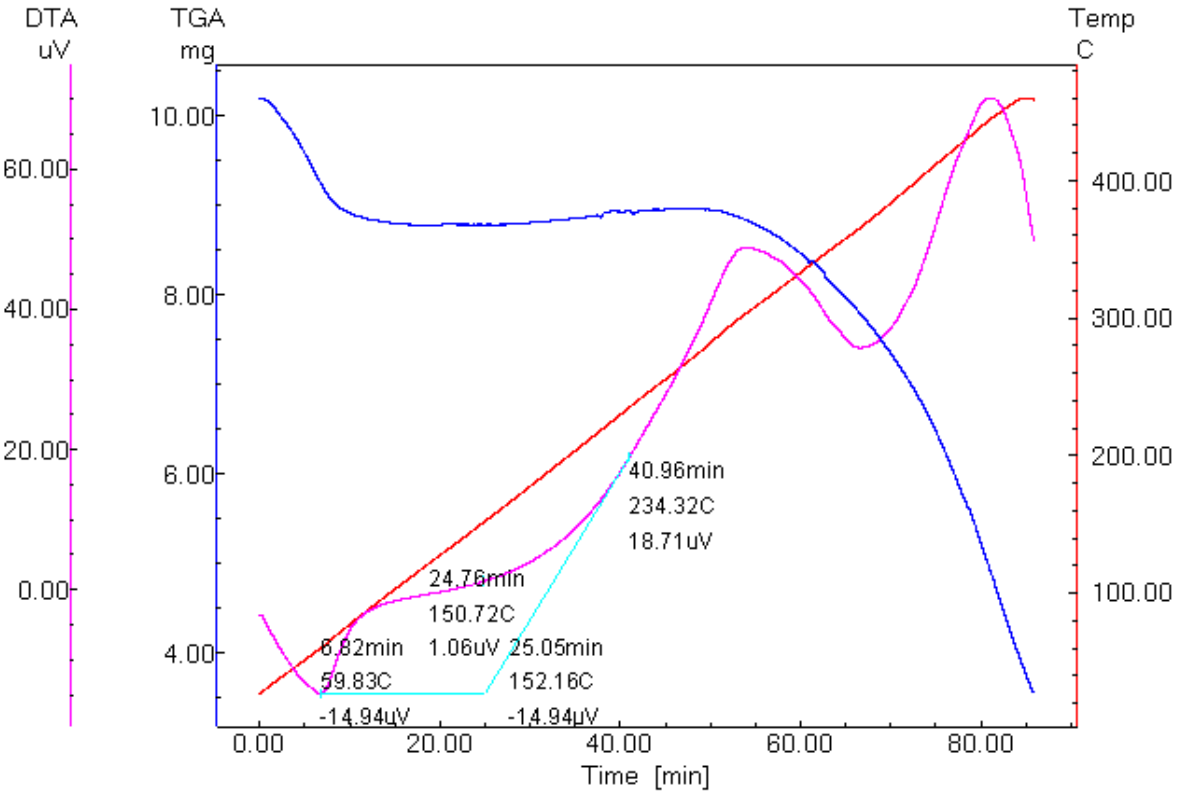

Fig A25 DTA – TG curve of MCL-8

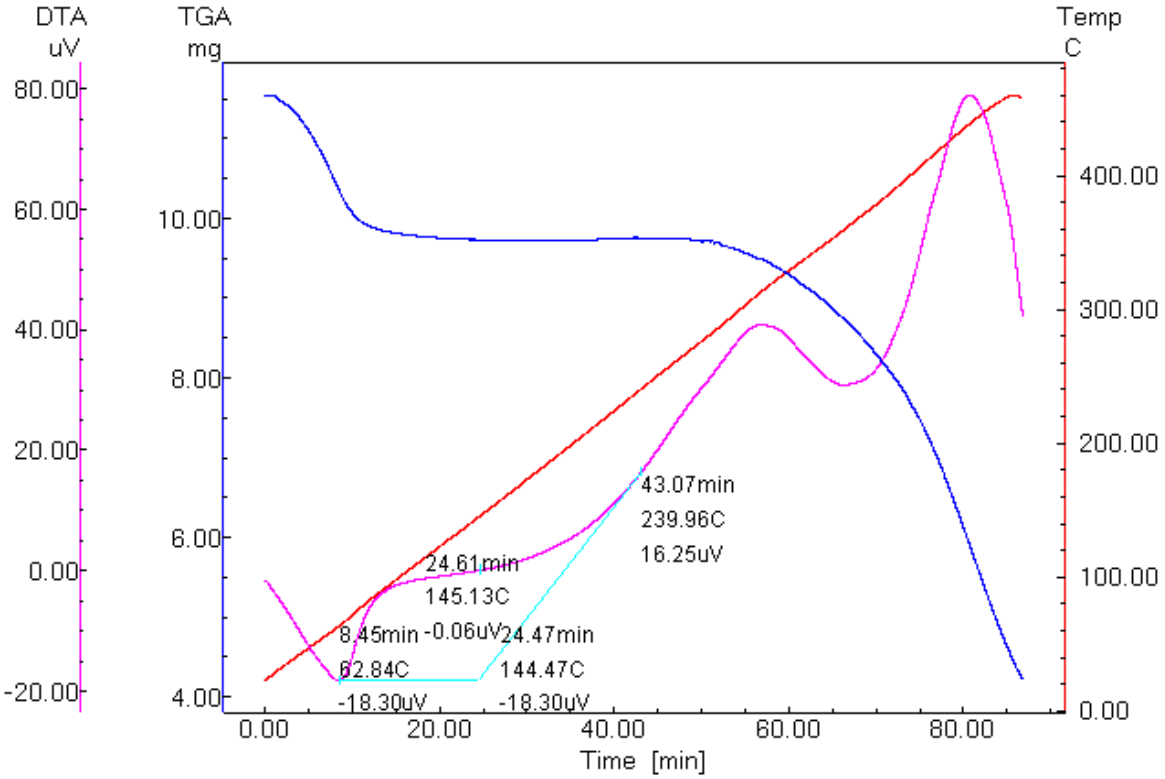

Fig A26 DTA – TG curve of NCL-1

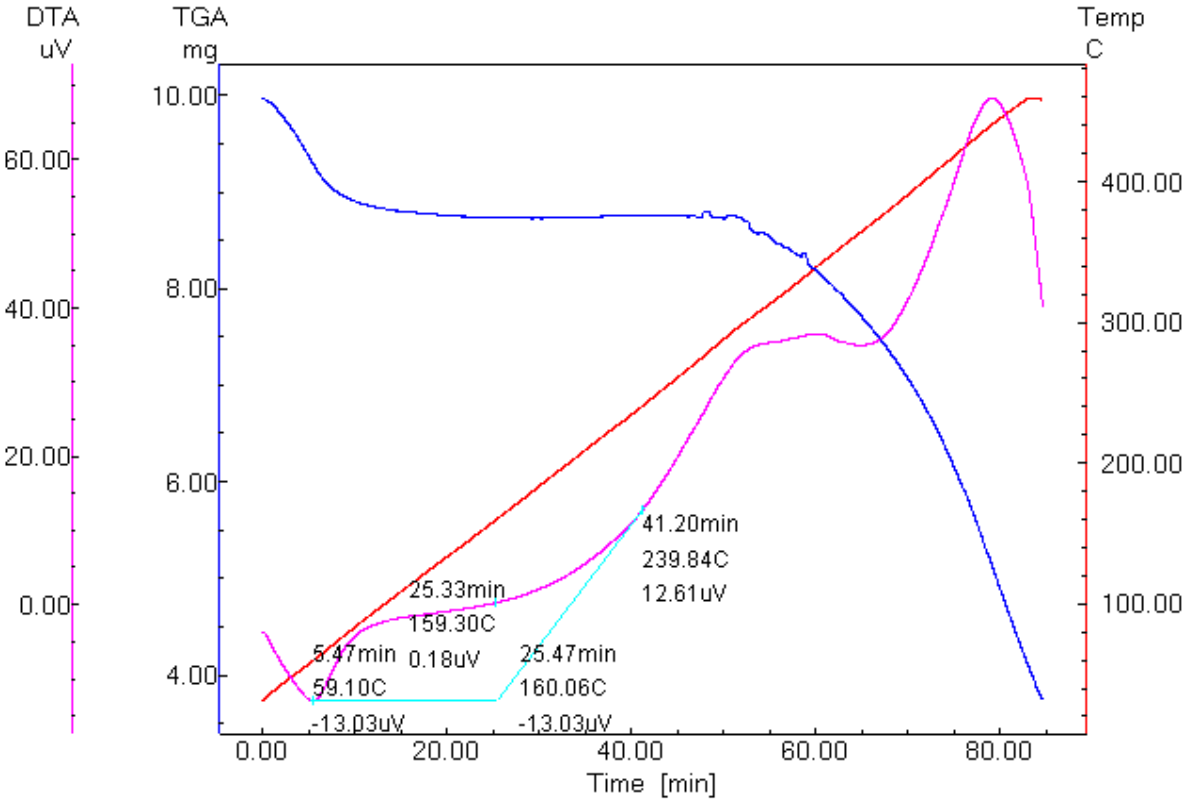

Fig A27 DTA – TG curve of NCL-2

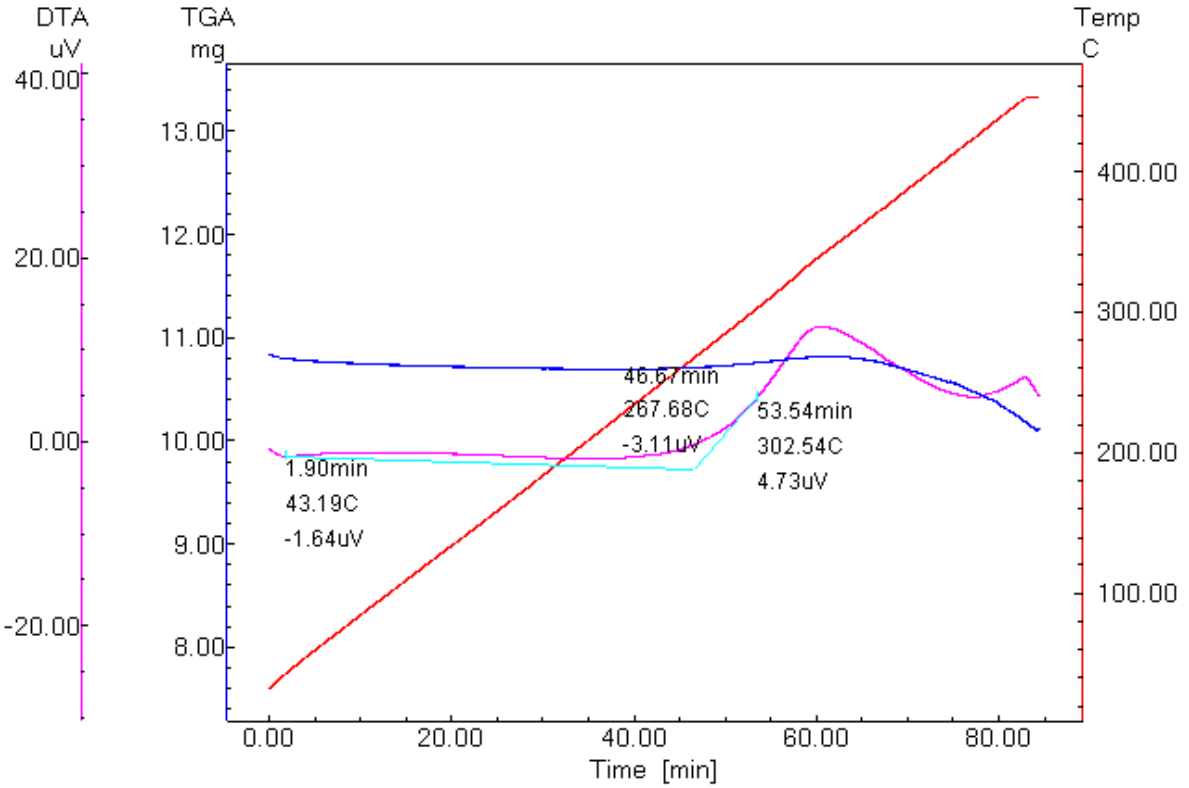

Fig A28 DTA – TG curve of IISCO-1

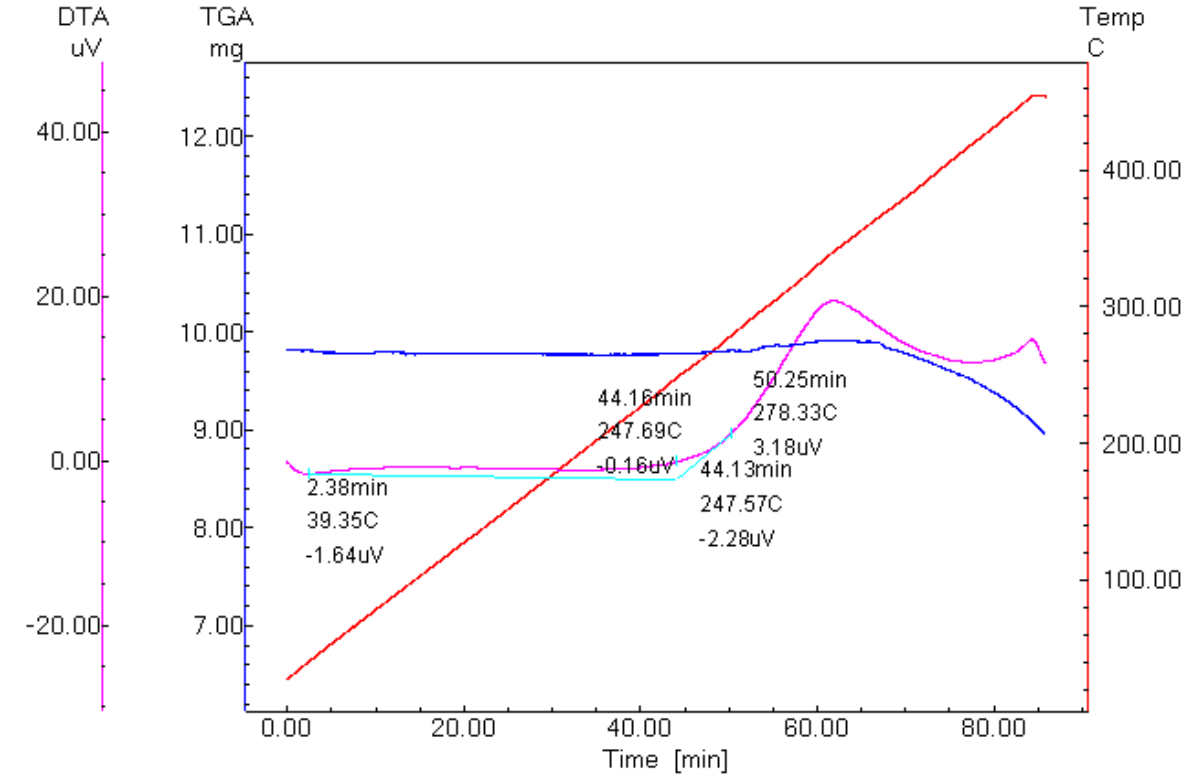

Fig A29 DTA – TG curve of IISCO-2

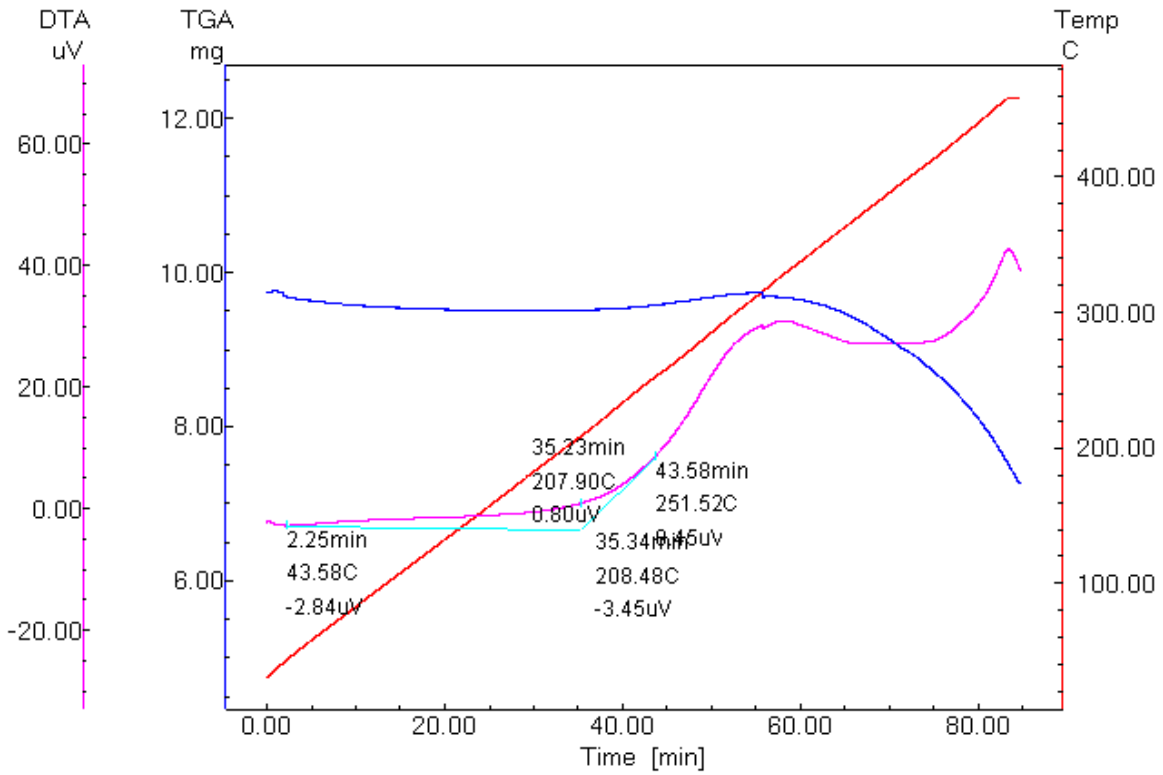

Fig A30 DTA – TG curve of IISCO-3

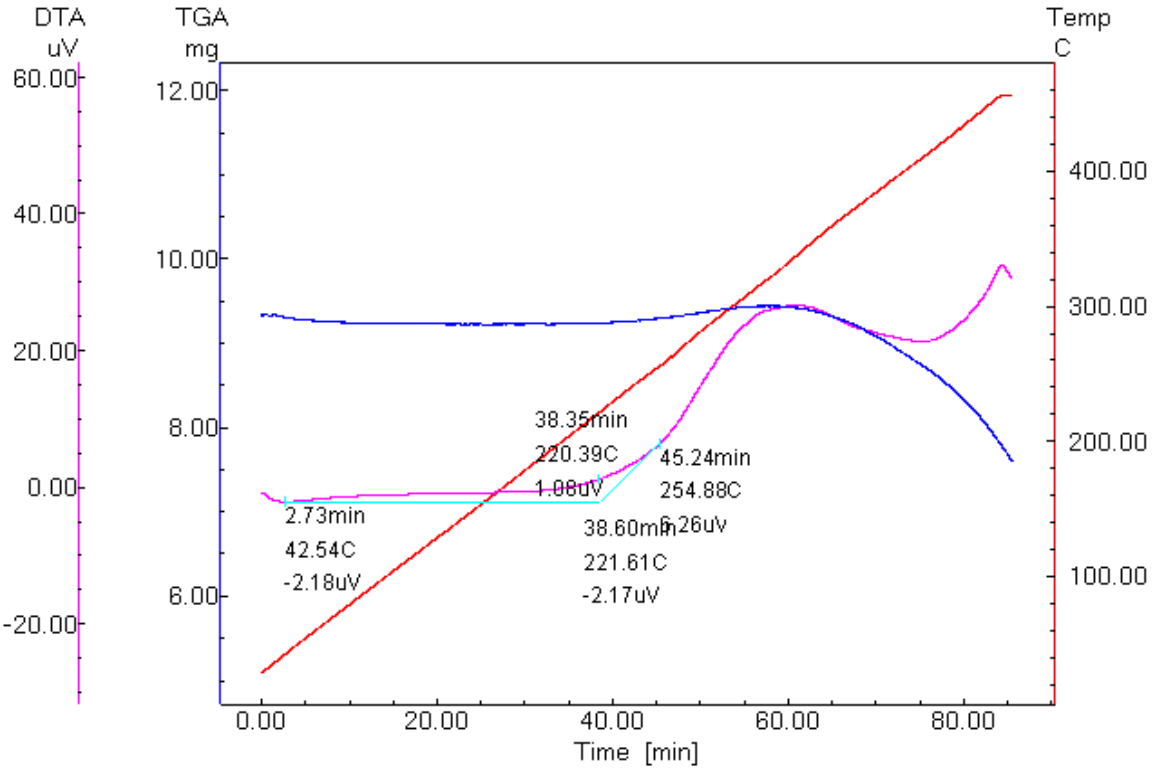

Fig A31 DTA – TG curve of IISCO-4

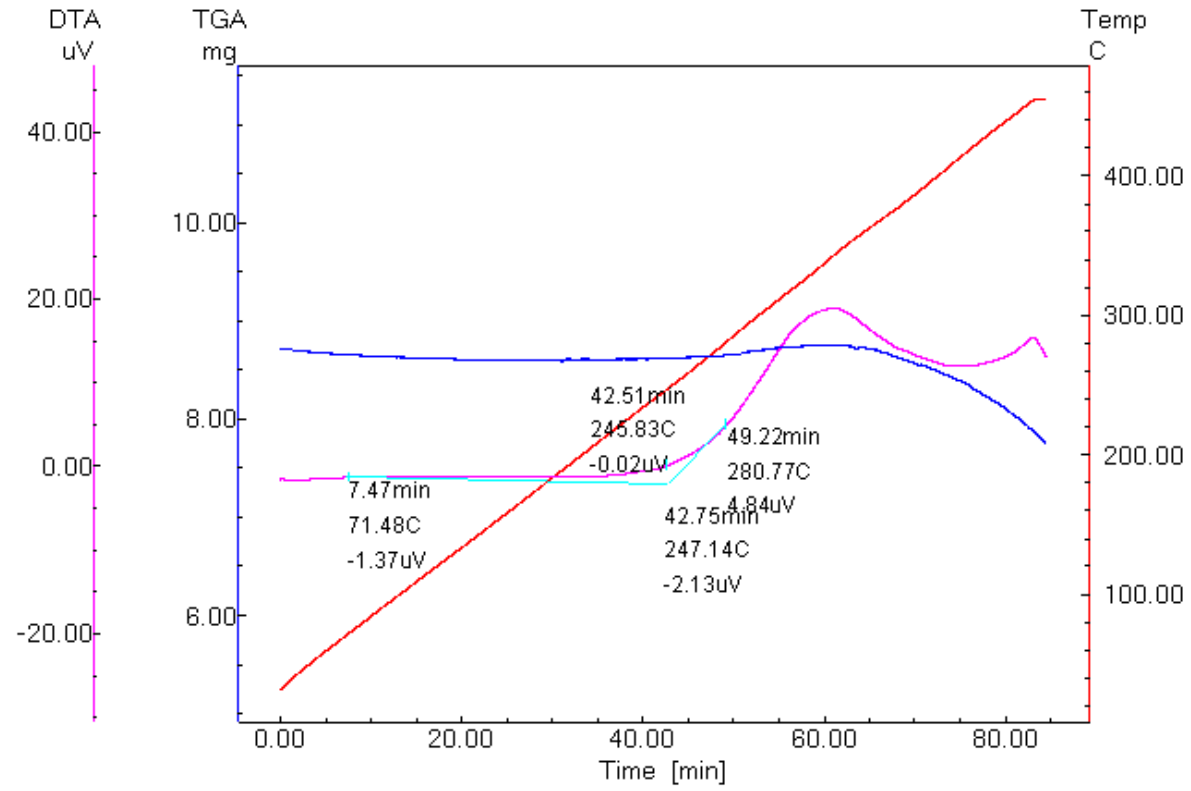

Fig A32 DTA – TG curve of BCCL-1

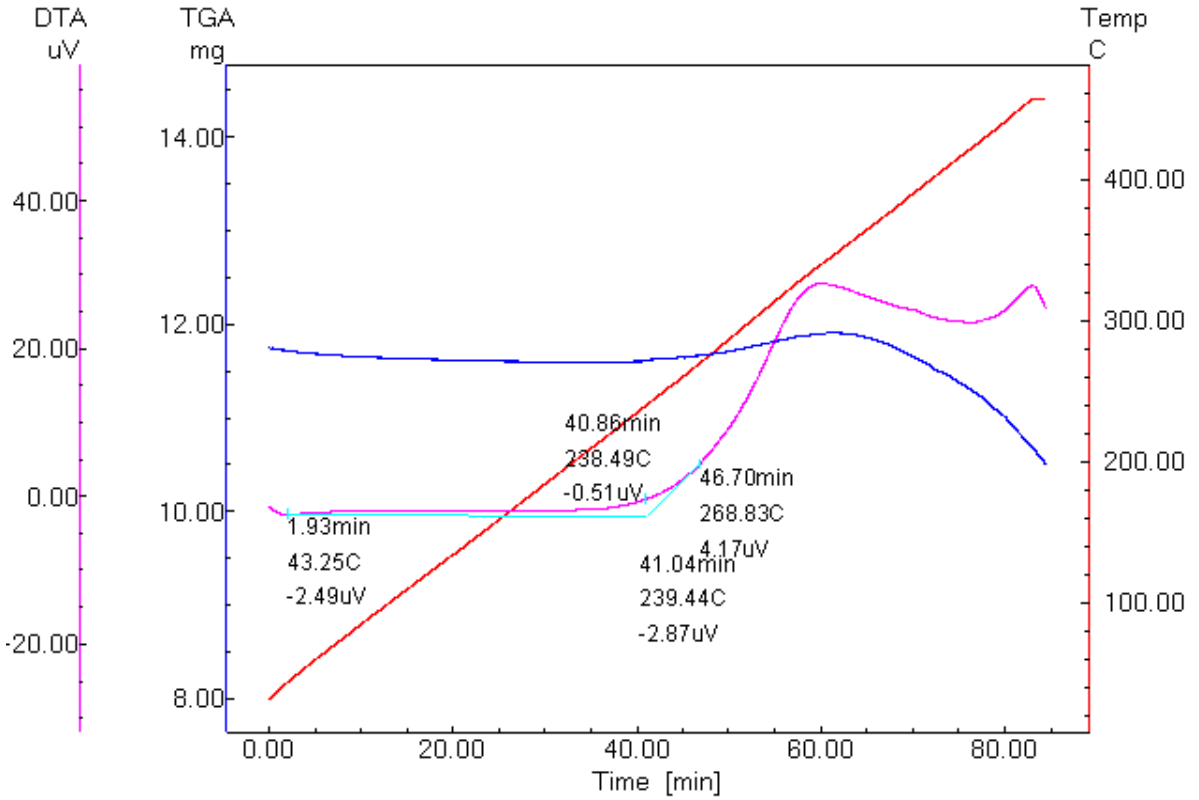

Fig A33 DTA – TG curve of TISCO-1